Water-window x-ray microscopy with laboratory sources

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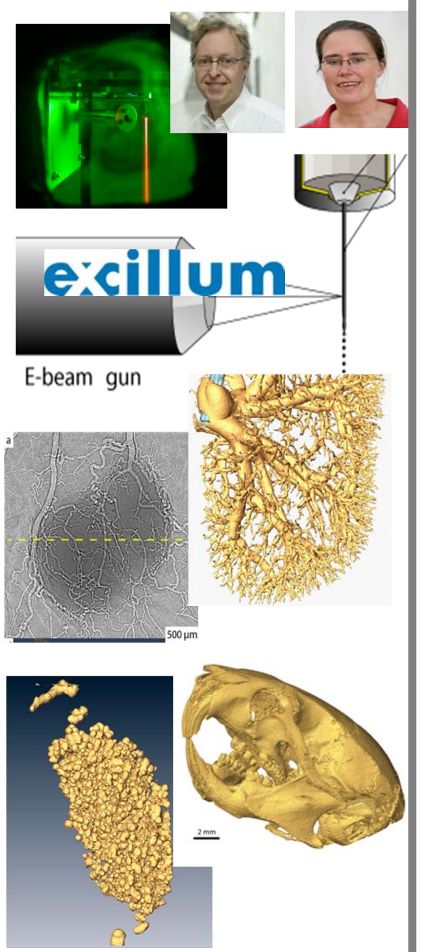
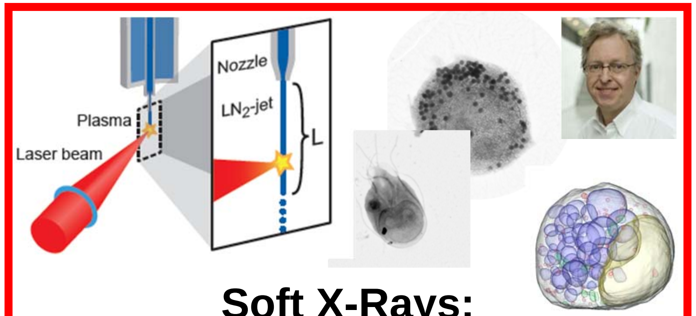


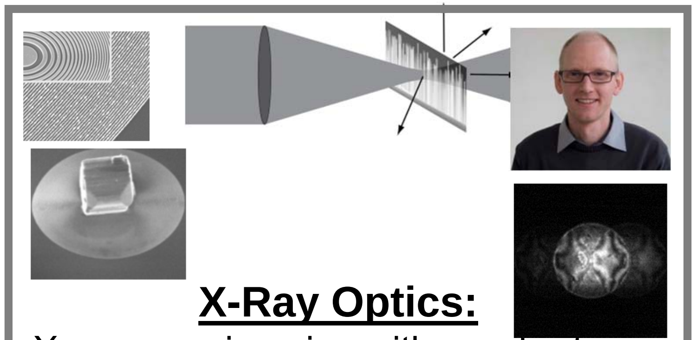
Hard X-Rays:

Sources, biomedical phase imaging



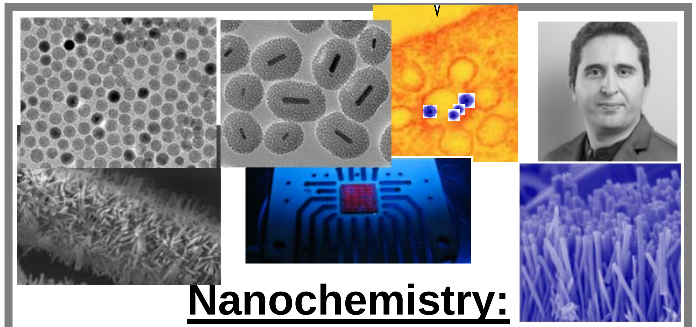
Soft X-Rays:

Sources, microscopy



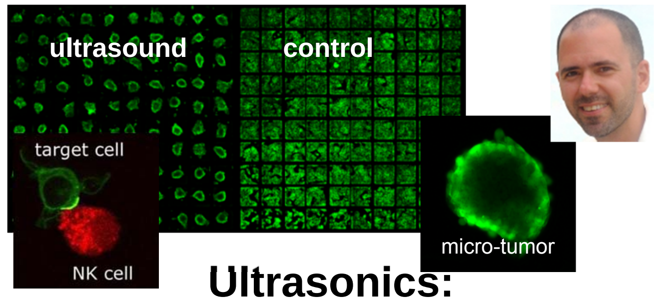
X-Ray Optics:

X-ray nano imaging with synchrotrons



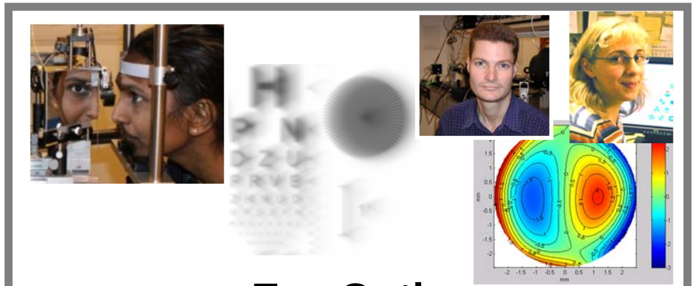
Nanochemistry:

Synthesis, analysis and applications



Ultrasonics:

Microfluidics, cell bio & bio analytics

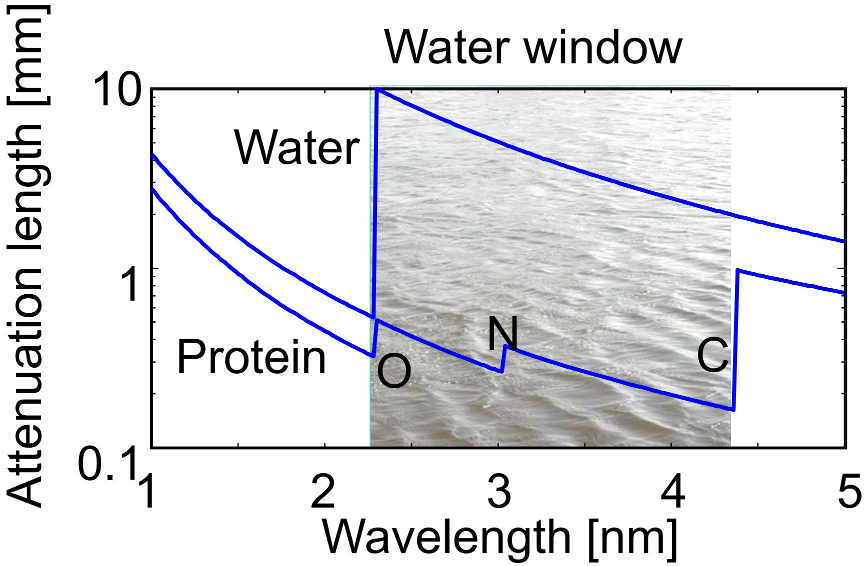


Eye Optics:

Peripheral vision, adaptive optics

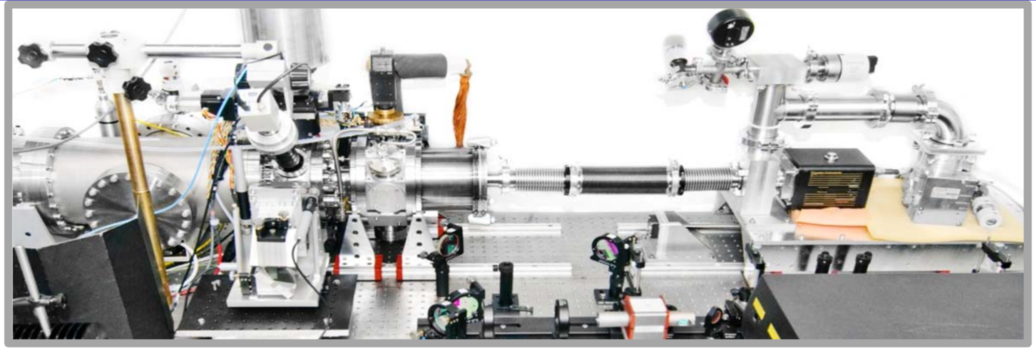
Teaching & Technical

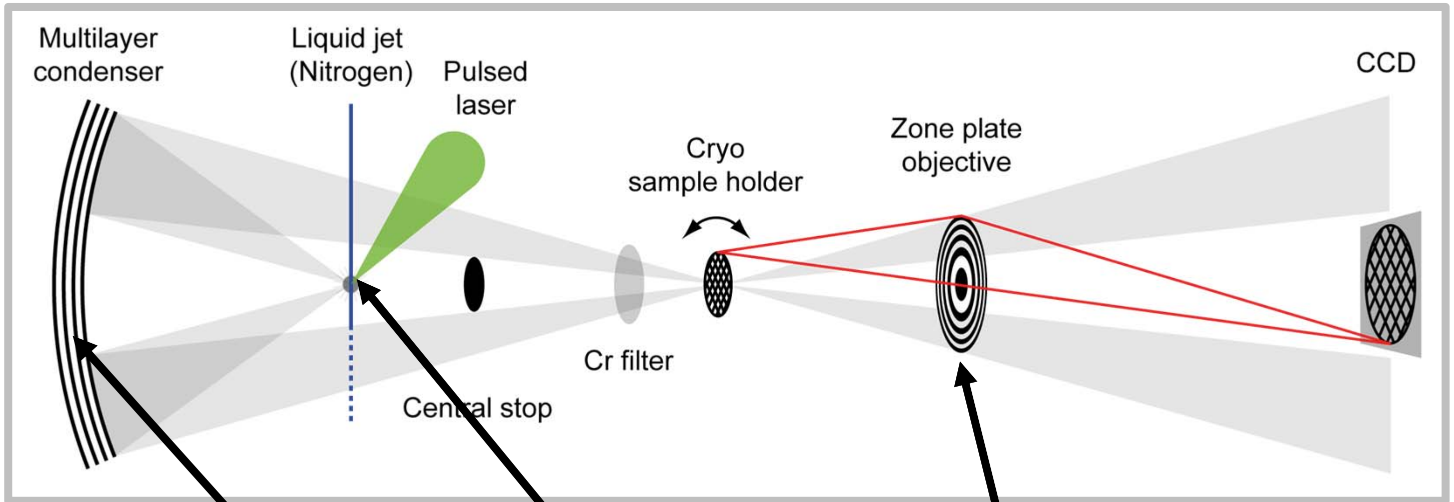
Water-window x-ray microscopy



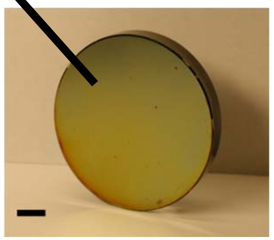
+ Resolution: 0.61λ/NA
+ Natural contrast for wet or frozen specimen
+ Possibility to study thick objects (approx. 10 μm water).
- Lack of compact high-brightness sources
- Inefficient optics

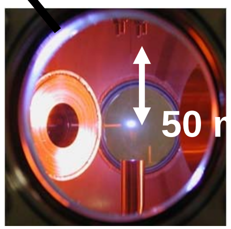
The Stockholm laboratory water-window x-ray microscope

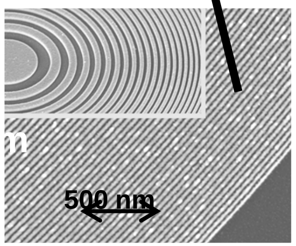




<u>Normal-incidence multilayer mirrors</u> as condensors







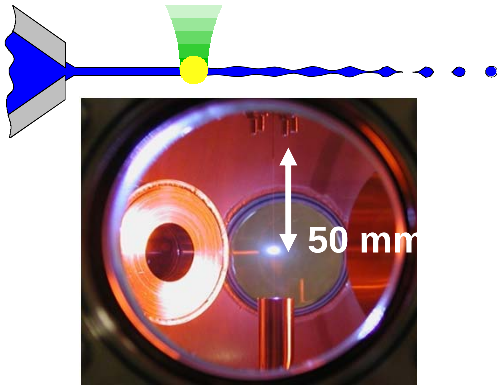
<u>Micro zone plates</u> for high-resolution imaging

Berglund et al, J. Microsc. (2000), Takman et al, J. Microsc. (2007), Bertilson et al, Opt Expr (2011); Bertilson et al Opt. Lett (2011) etc

In brief:

Liquid-jet/droplet laser-plasma sources

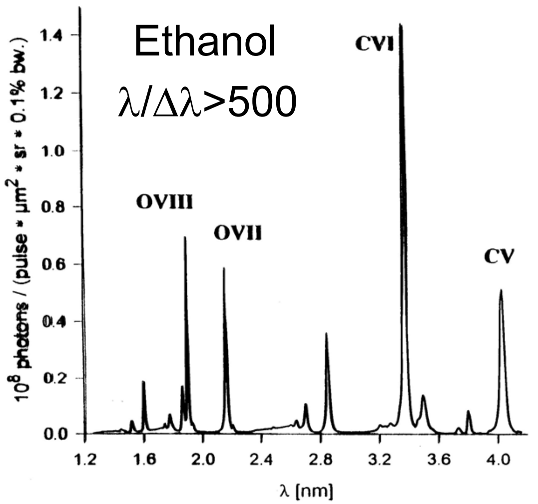
Principles



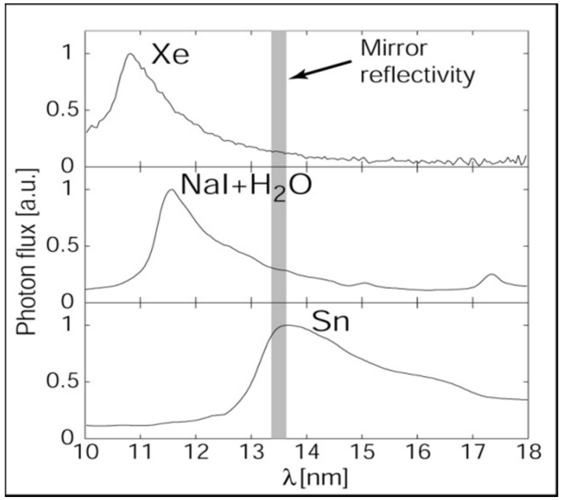
+ Negligible debris
+ High rep.-rate operation
+ Full-day operation possible
+ Tailored spectral emission
+ High-power operation possible

Rymell et al, Opt. Commun. (1993)

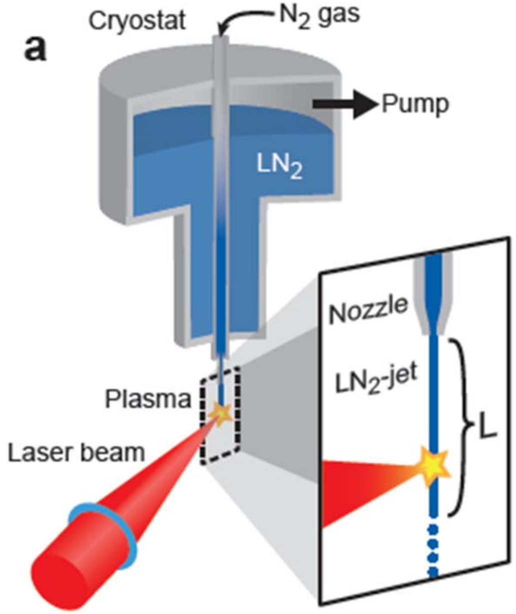
λ≈2-4 nm: Water-window

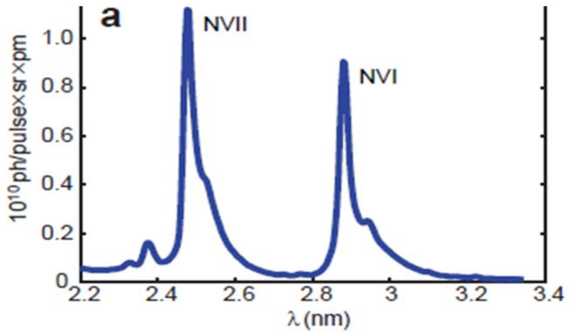


λ=13 nm: EUV Litho



λ≈2.4 nm Liquid nitrogen

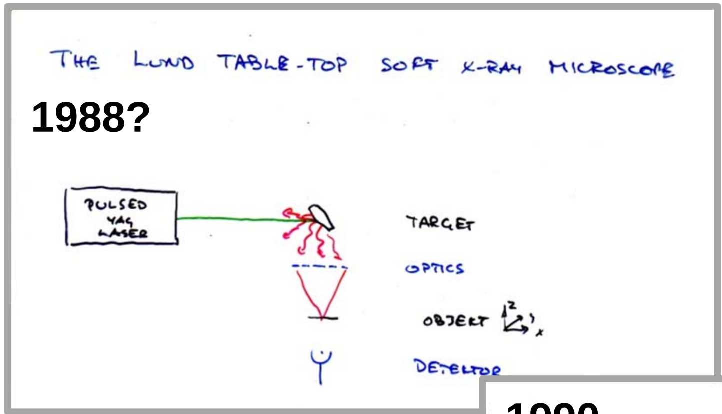




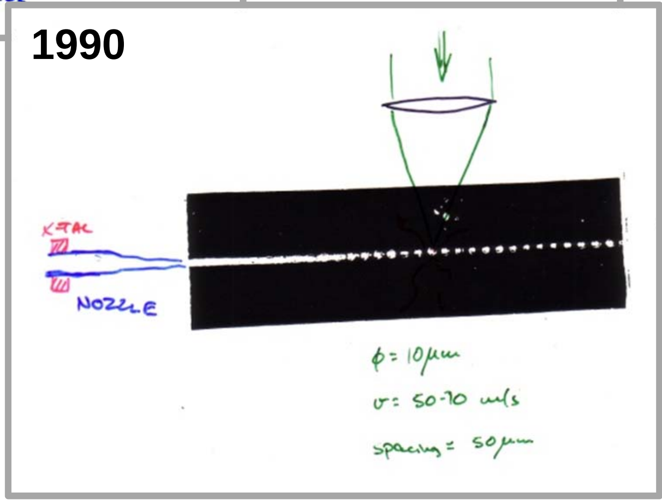
Rymell et al, APL (1995); Berglund et al APL (1997);Jansson et al, RSI (2005) ;Hansson et al, MNE (2001);Takman et al APL (2004) ;Martz et al, Opt. Lett. (2012)

Liquid-jet/droplet laser-plasma sources:

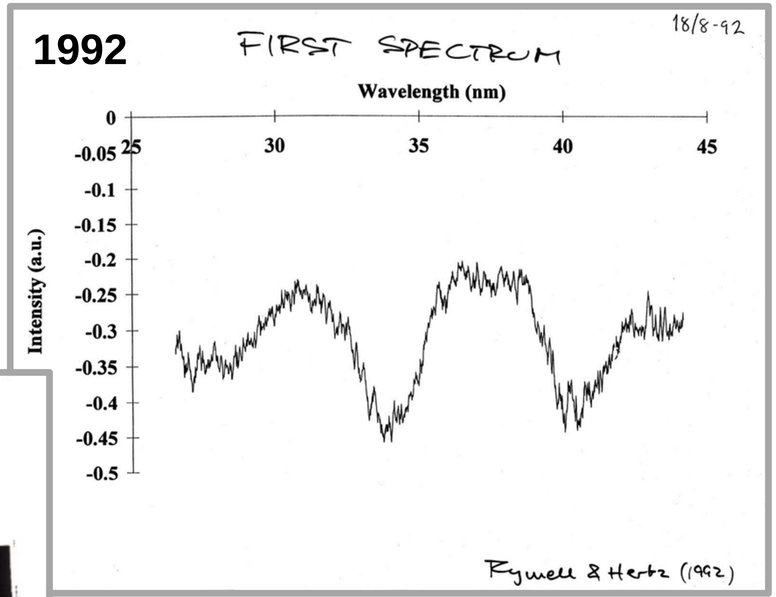
Early history



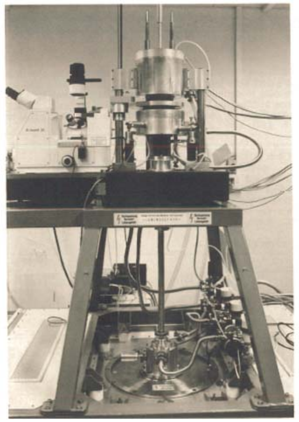
First XRM slide



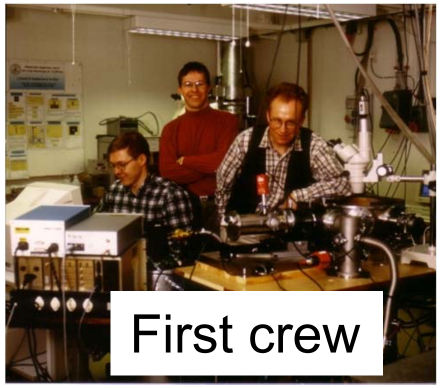
First liquid-droplet Laser-plasma slide



First spectrum



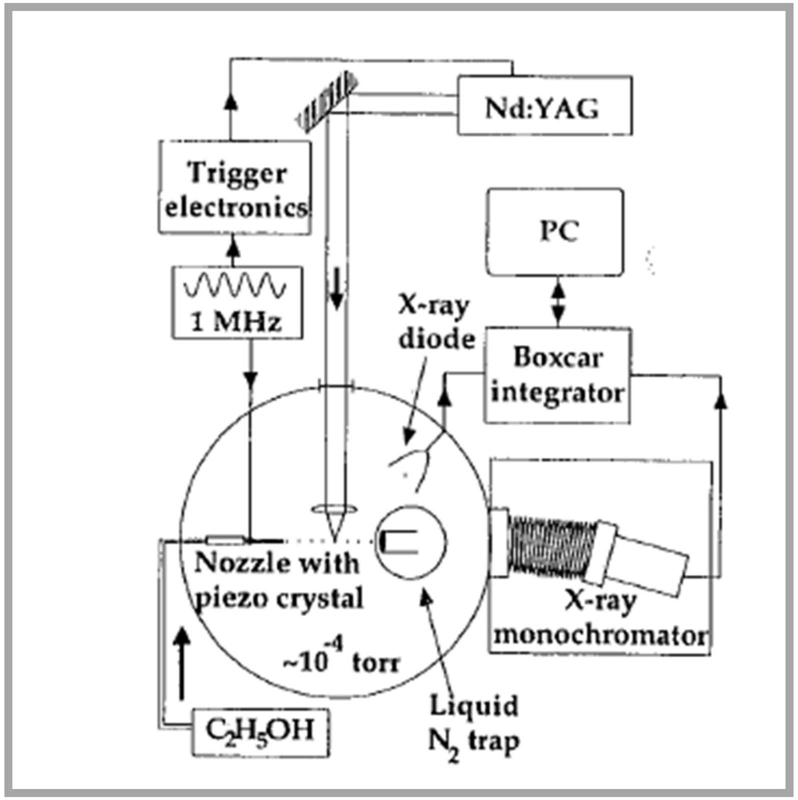
Cf. Göttingen

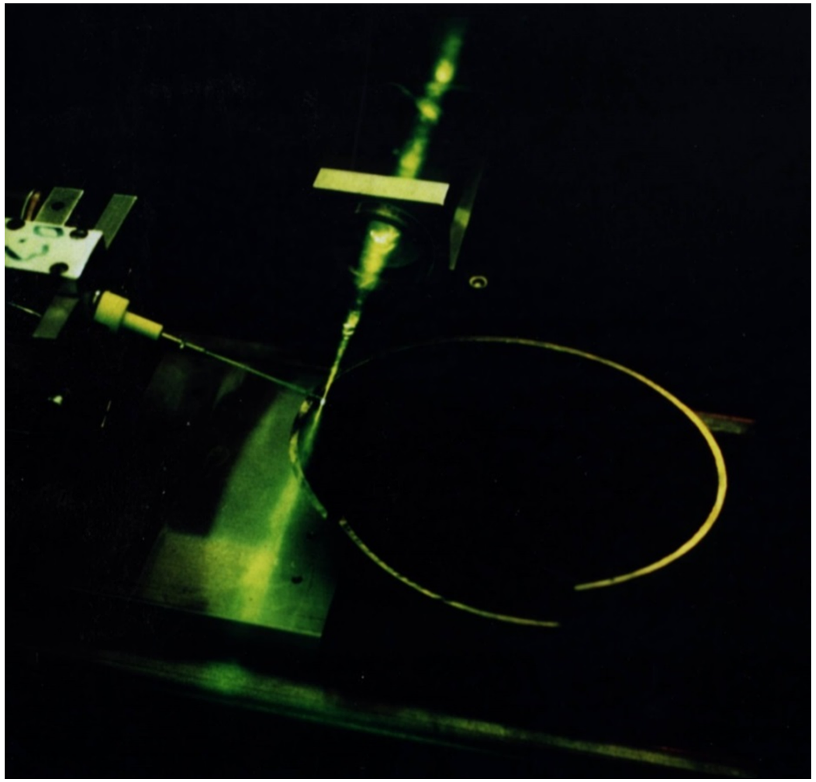


First crew

Liquid-droplet laser-plasma sources:

Early experimental arrangement

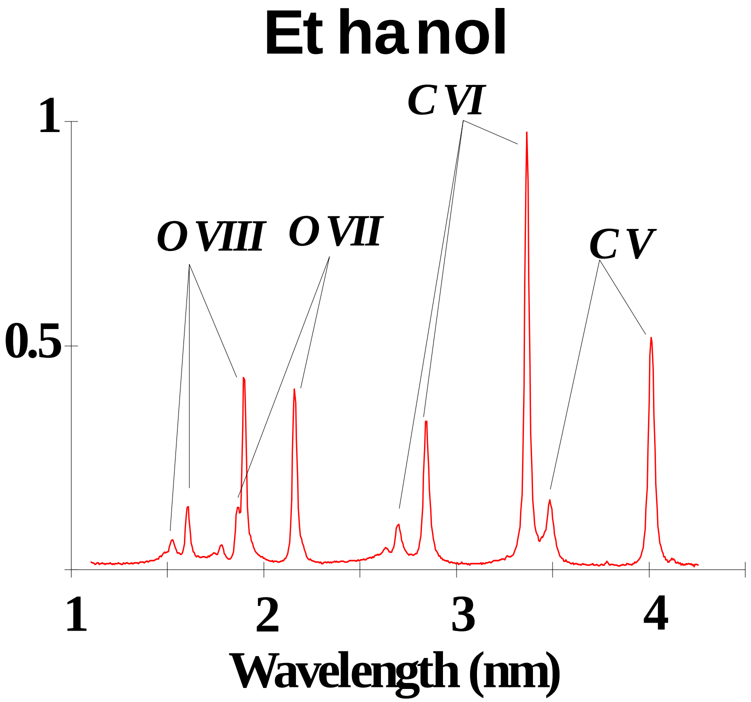


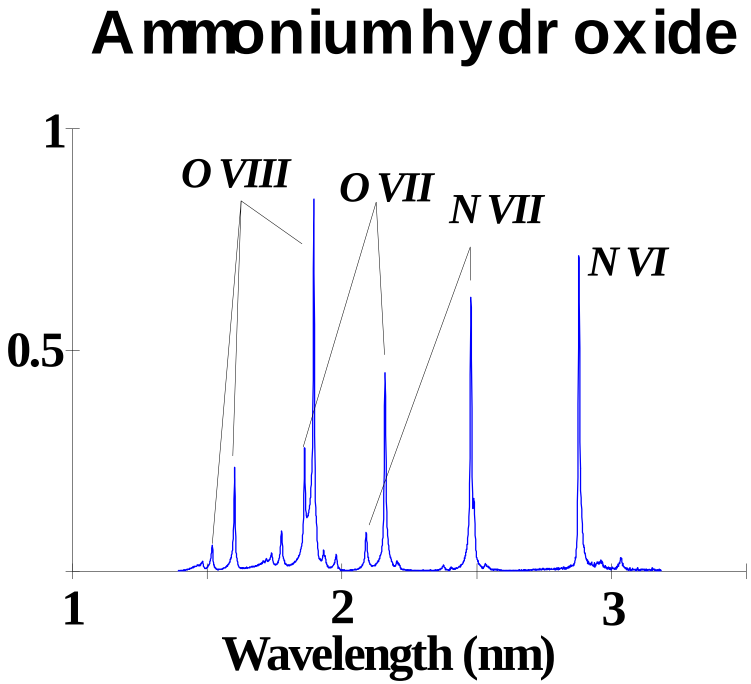


Rymell et al, Opt. Commun. (1993)

Liquid-jet/droplet laser-plasma sources:

Water-window x-ray emission

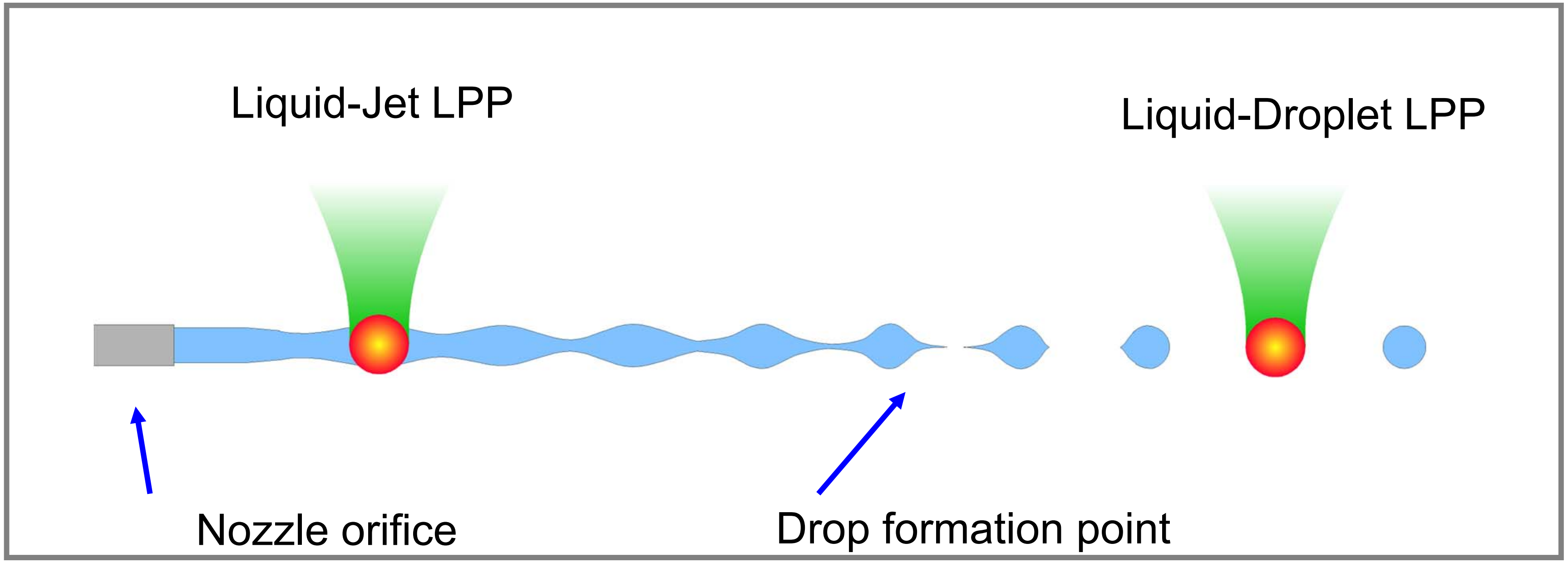




Typically 10^{12} ph/sr×line×pulse with 100 mJ, 100 ps SHG YAG

Rymell et al, Opt. Commun. (1993)
Rymell et al, APL (1995)

Liquid-jet/droplet laser plasmas

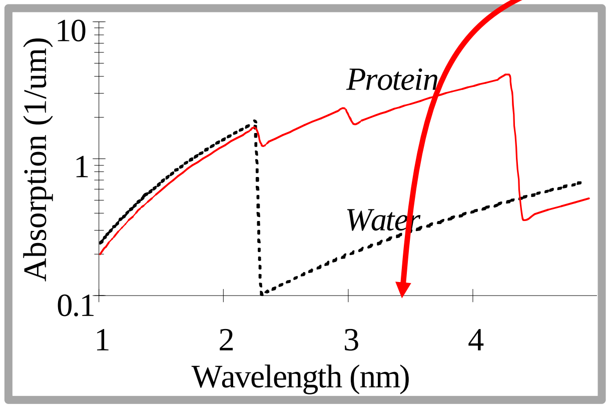


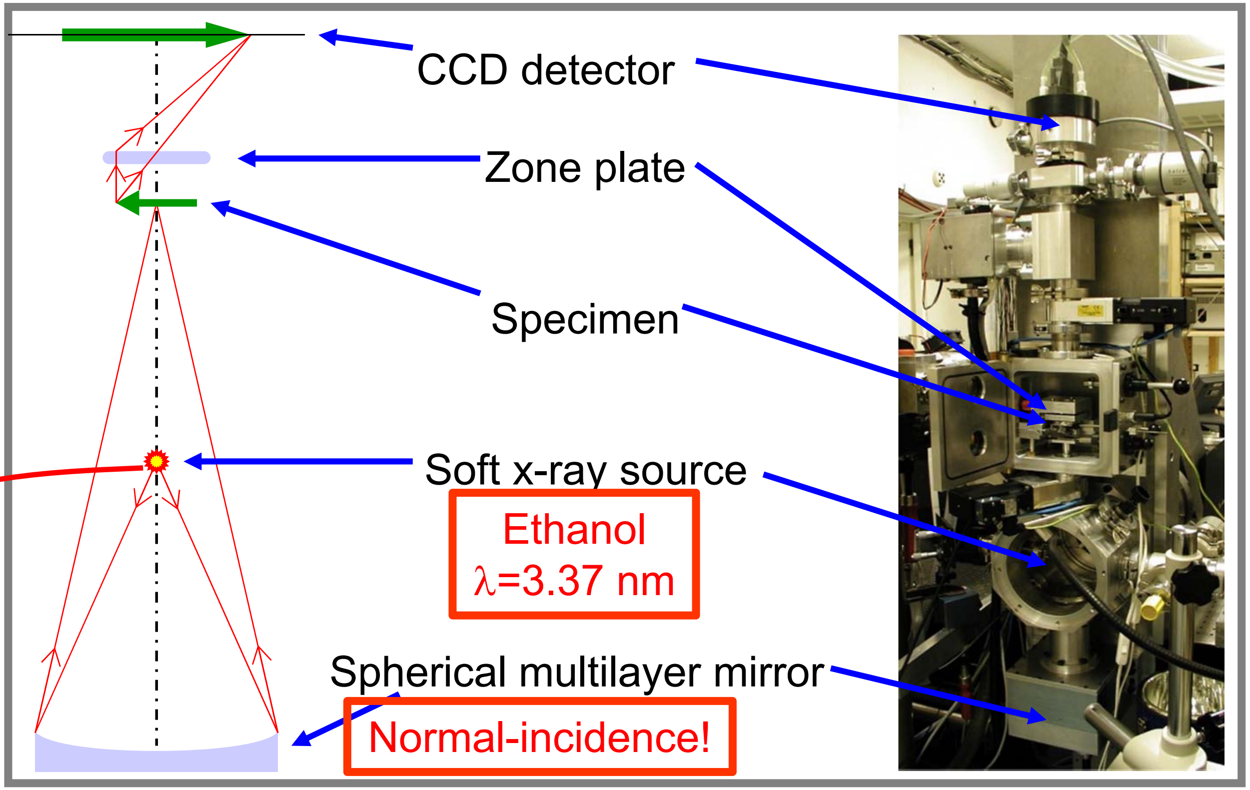
+ Negligible debris
+ High rep.-rate operation possible
+ Full-day operation possible
+ Tailored spectral emission
+ High-power operation possible

Rymell et al, Opt. Commun. (1993)
Malmqvist et al, RSI (1996)

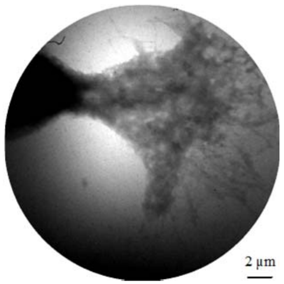
First compact laser-plasma x-ray microscope

- Resolution
- Contrast
- Thick samples
- Wet environment





Diatom



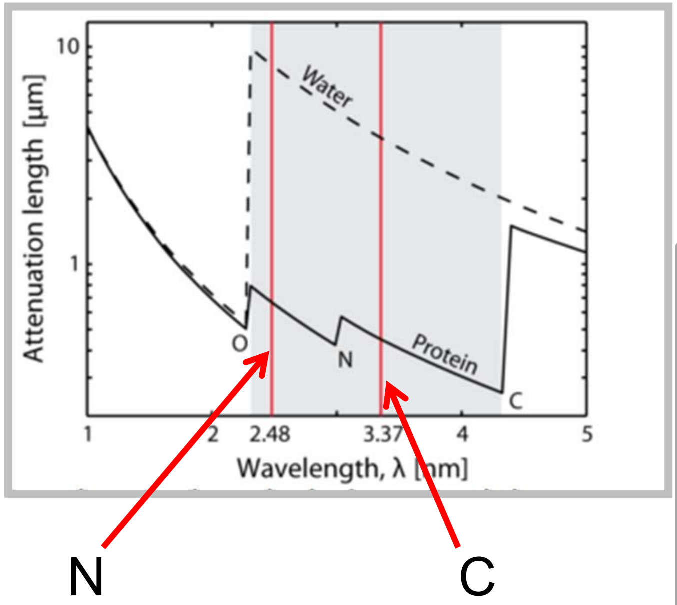
Fixed COS-7

Berglund et al, J. Microsc. (2000)

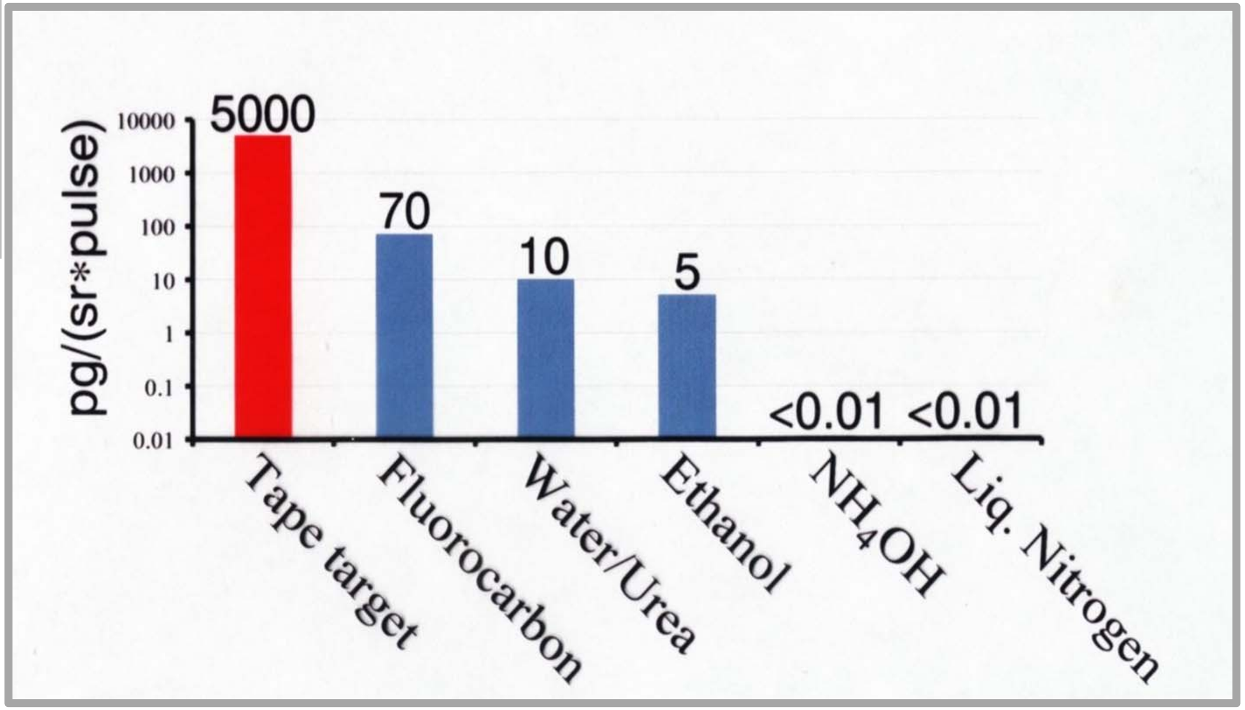
Problems:

Transmission & Debris

Transmission



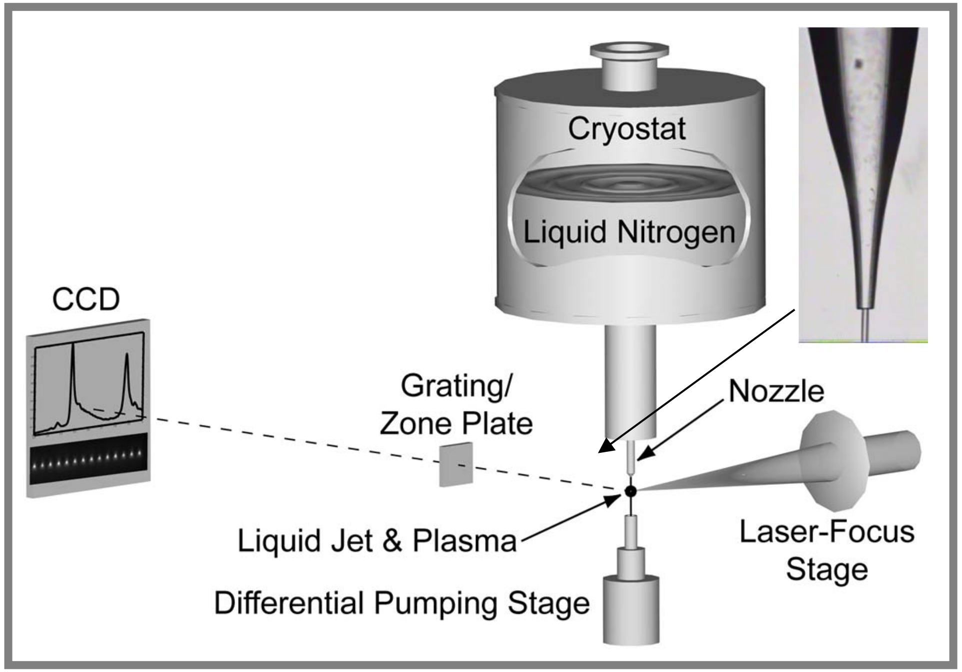
Debris deposition

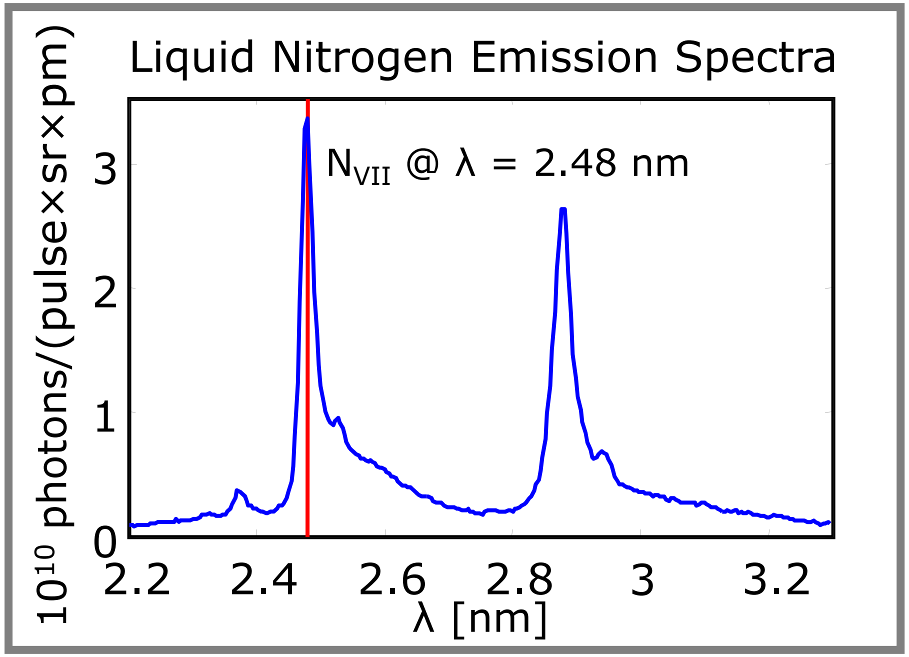


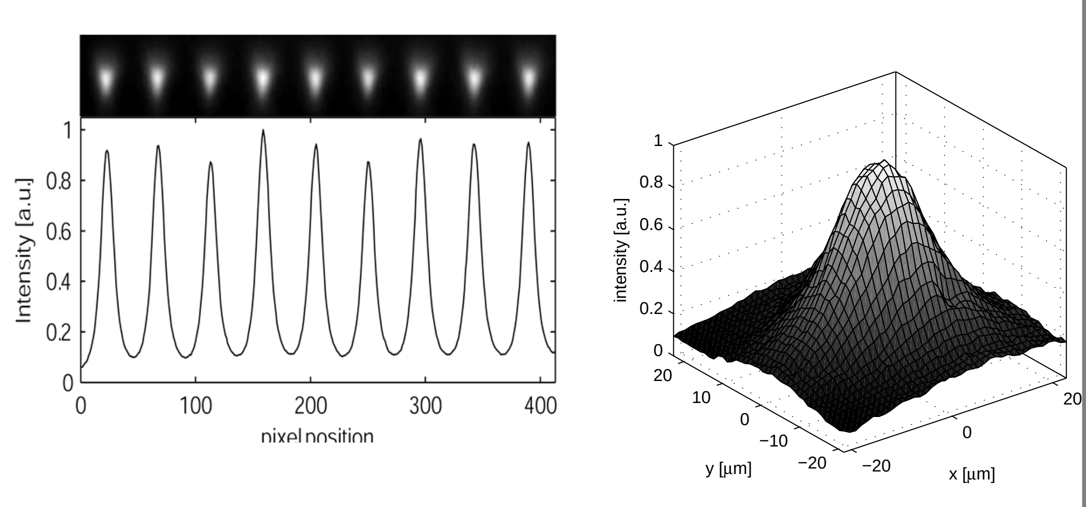
Rymell et al, RSI (1995), Berglund et al

Laboratory water-window laser-plasma sources:

The liquid-nitrogen-jet source







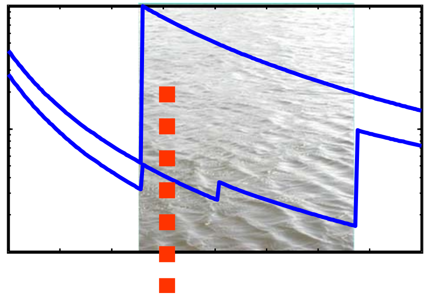
Laser: 20 W, 100 Hz, 3 ns

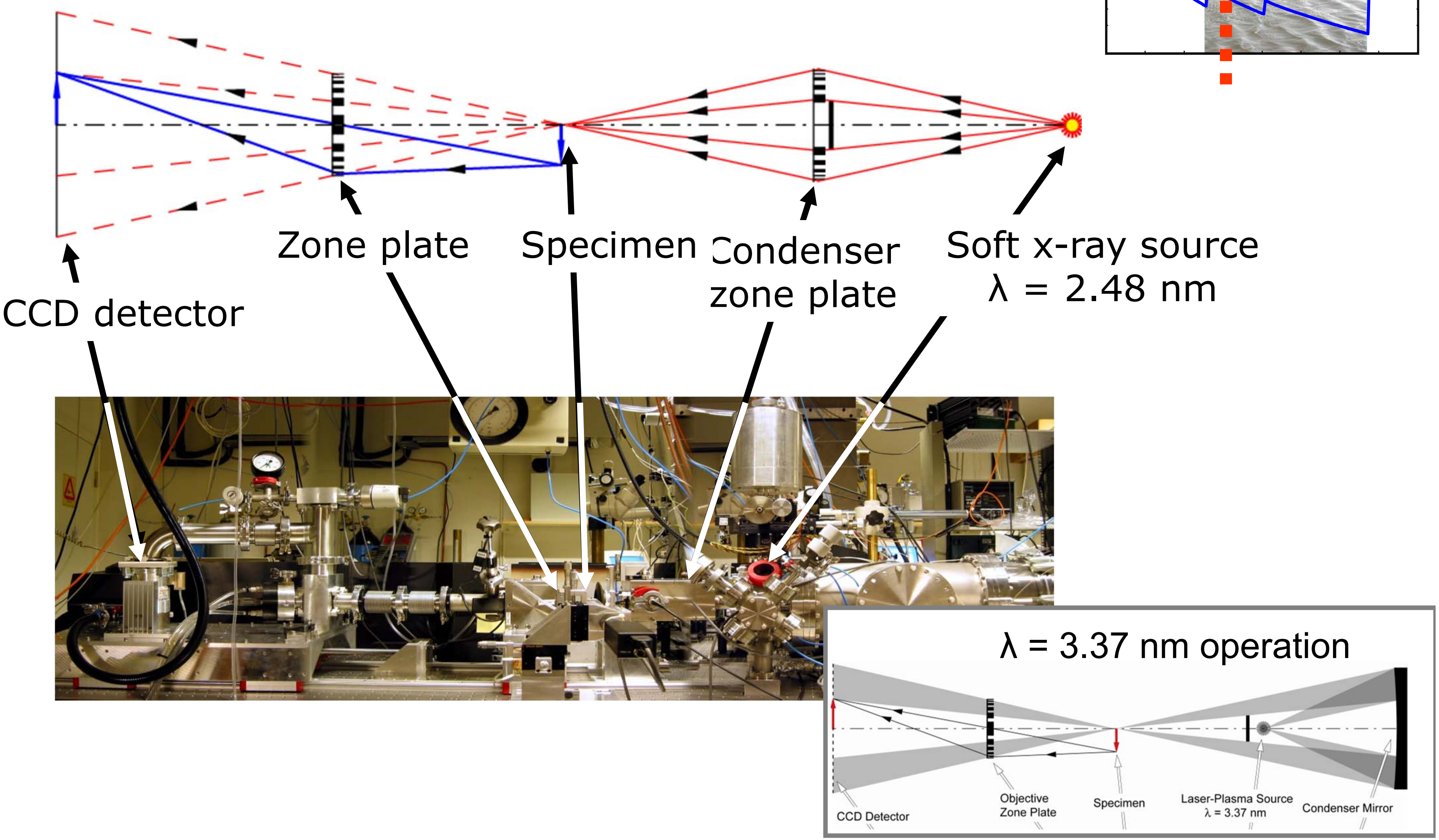
Flux: 1×10^{12} ph/pulse×sr×line
Stability: ±2 µm
Brightness: 4×10^{8} photons/(pulse×sr×µm^2×line)
Illumination Uniformity: 10%

Berglund et al, RSI (1998), Jansson et al RSI (2005)

Laboratory water-window microscopy

@ λ=2.48 nm

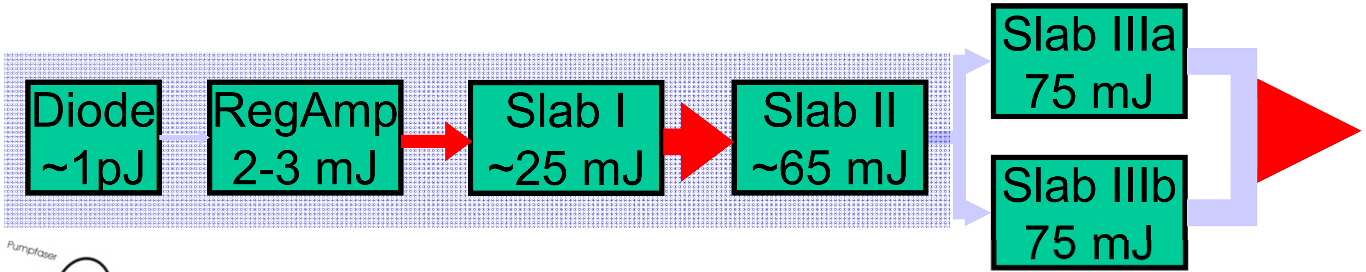


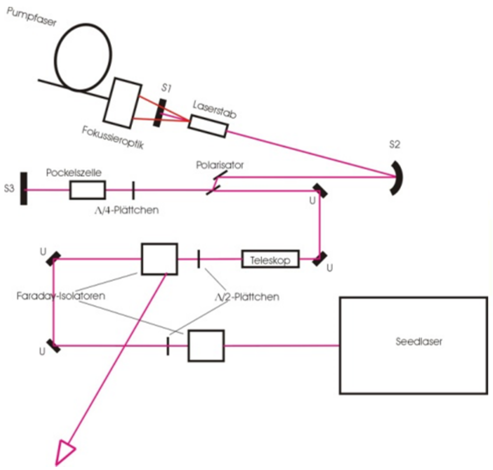


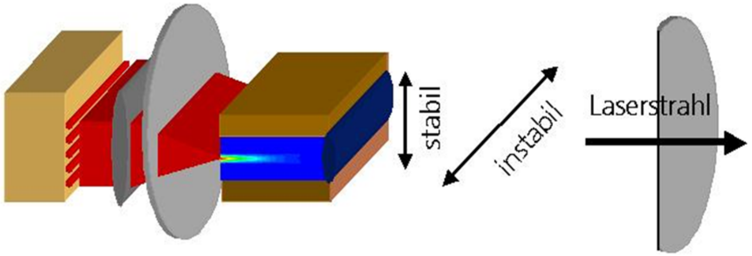
Berglund et al, J. Microsc. (2000), Johansson et al, RSI (2002) Takman et al, J. Microsc. (2007)

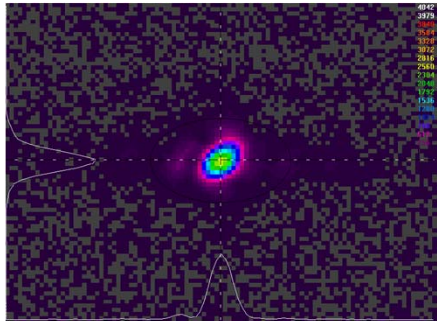
Next-generation liquid-jet laser plasmas:

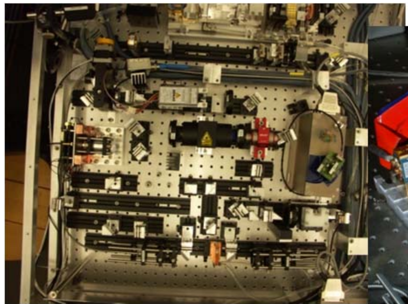
260 W, 800 ps, 2 KHz DPSS

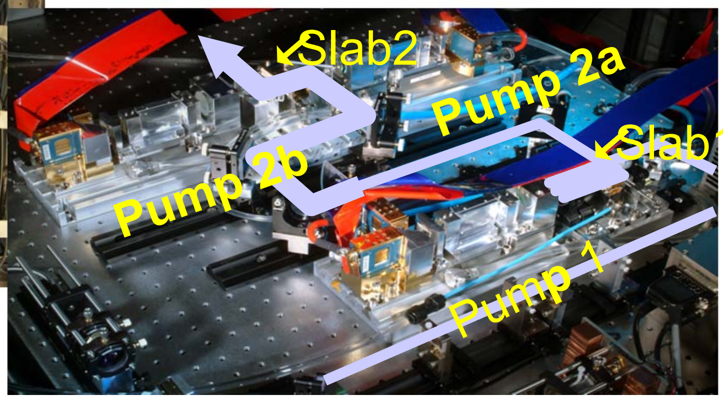


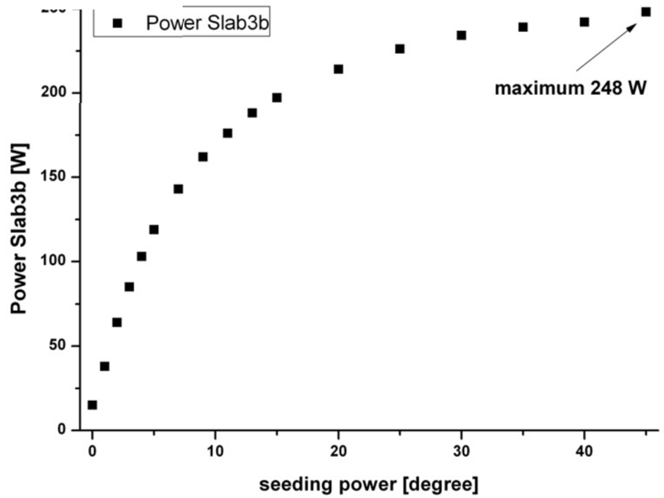










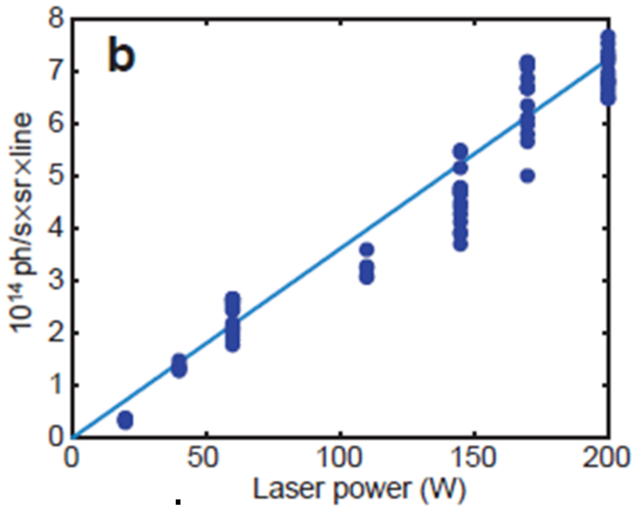


Thanks to Dominik Esser et al, FhG ILT, Aachen

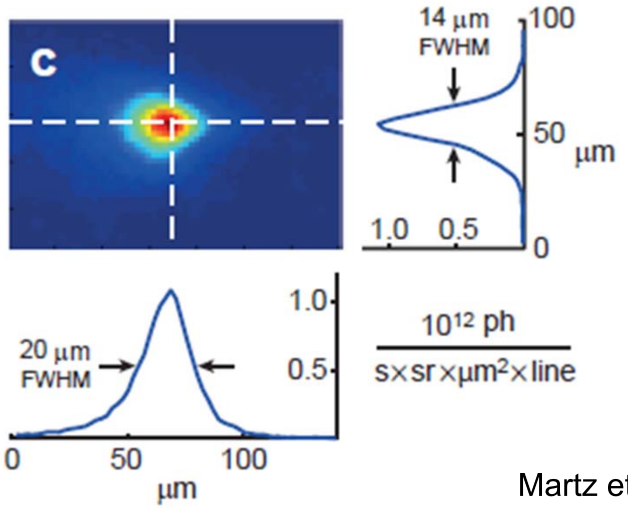
High average brightness:

200 W, 600 ps, 2 KHz DPSS laser plasma

Photon flux

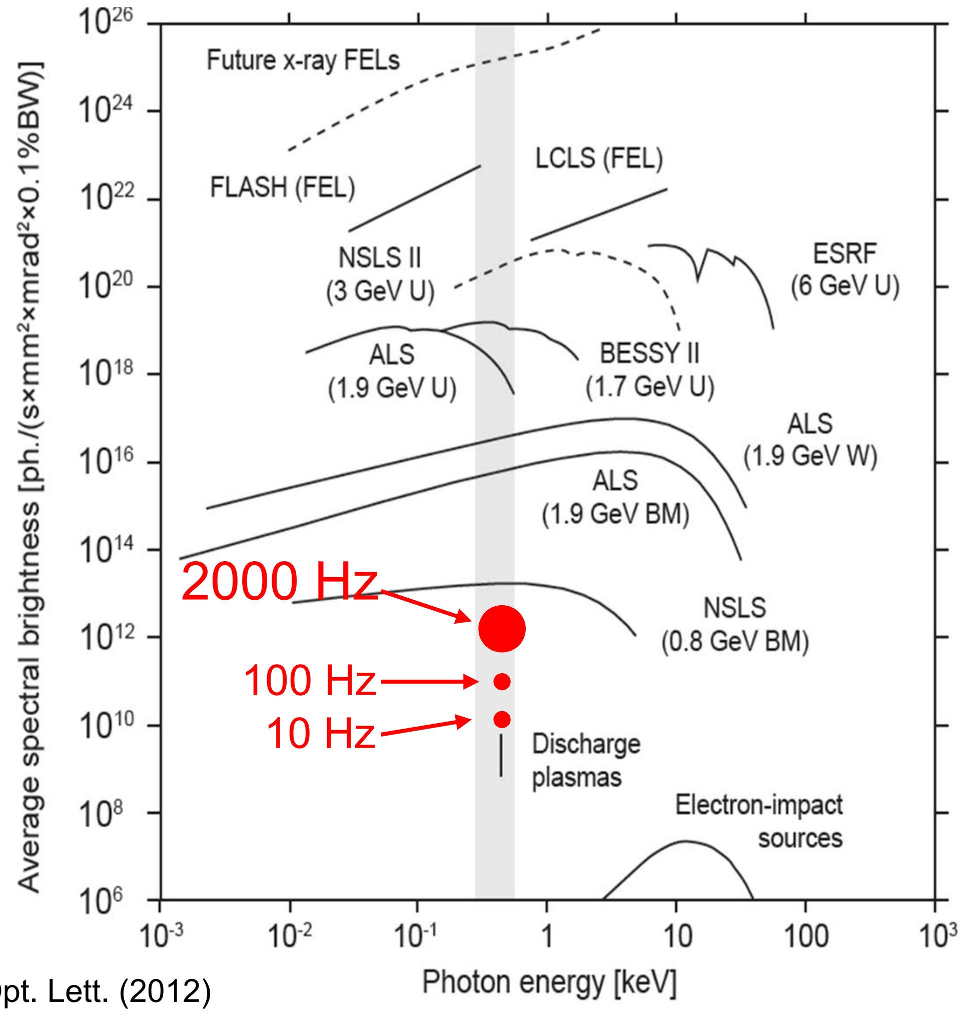


Source size



Spectral brightness:

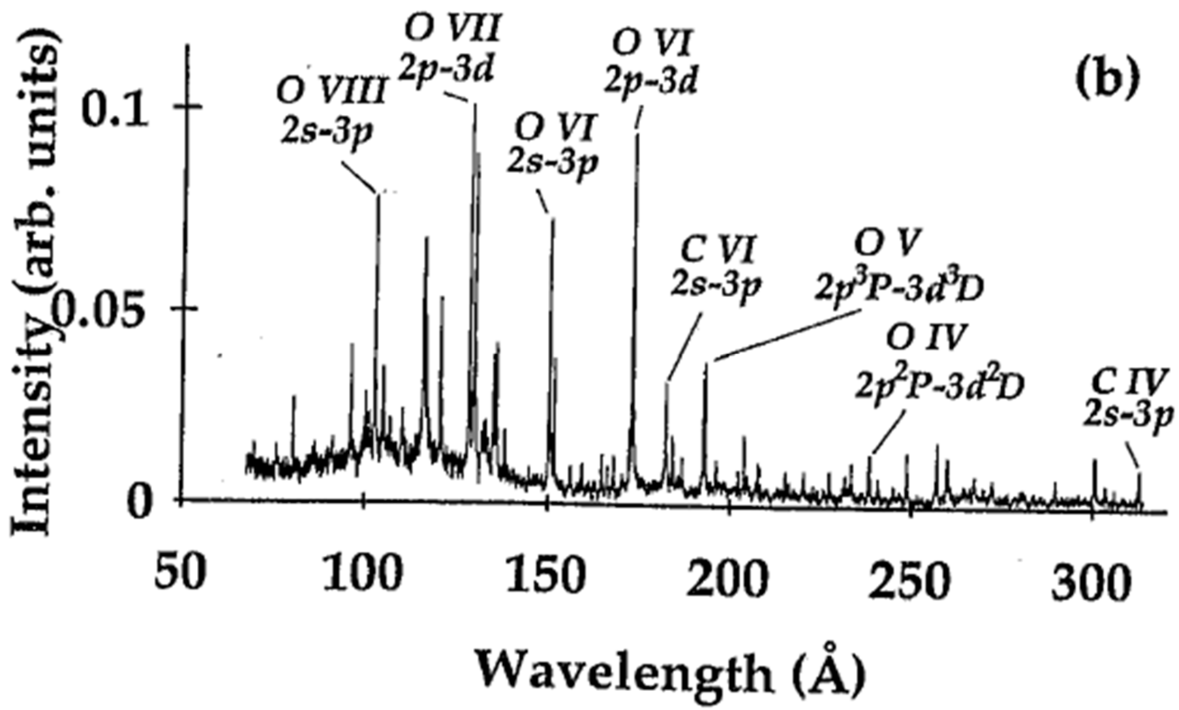
Lab source at early-bending-magn level!



Martz et al, Opt. Lett. (2012)

IN PARALLELL

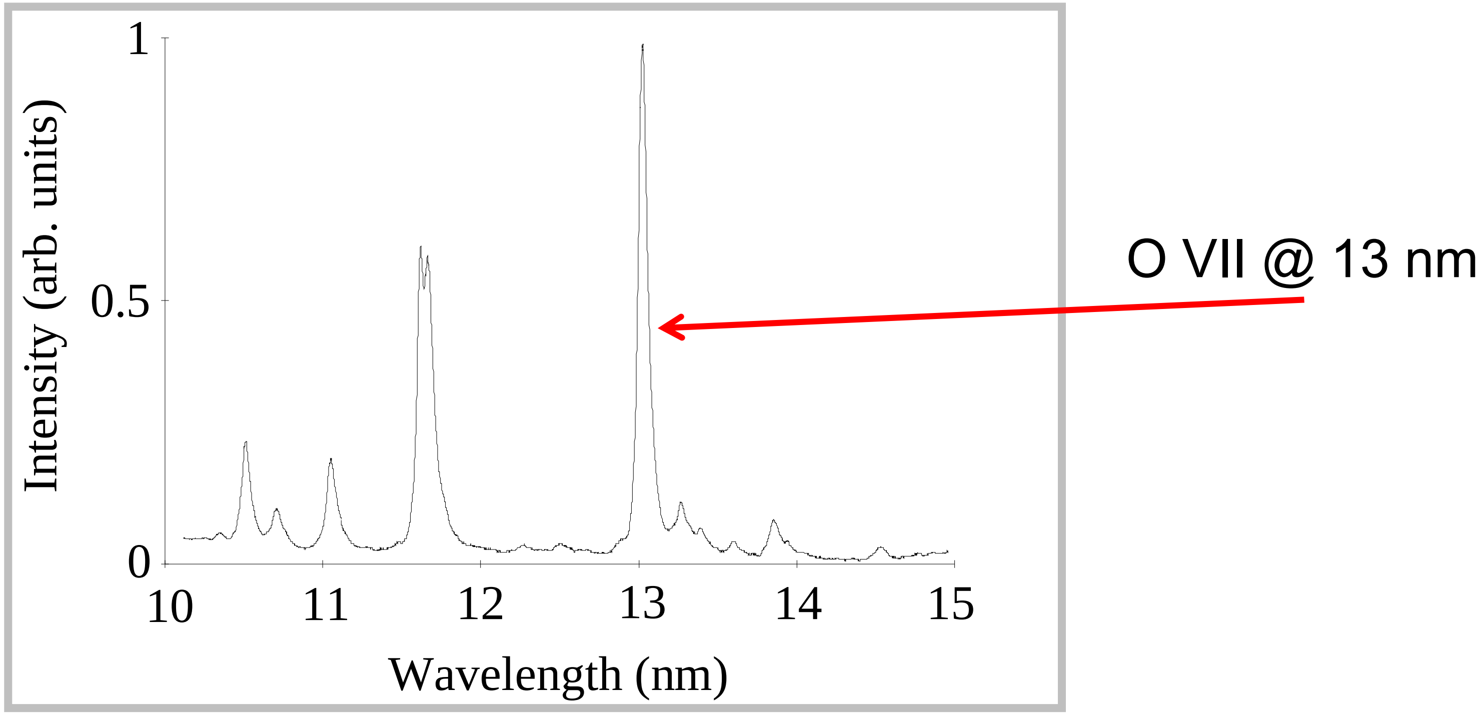
First liquid-droplet EUV source – oxygen @ 13 nm



Ethanol target
Nd:YAG 10 ns
Low debris

Rymell et al, Proc. XRM IV (1993)

Next liquid-droplet EUV source - water droplets



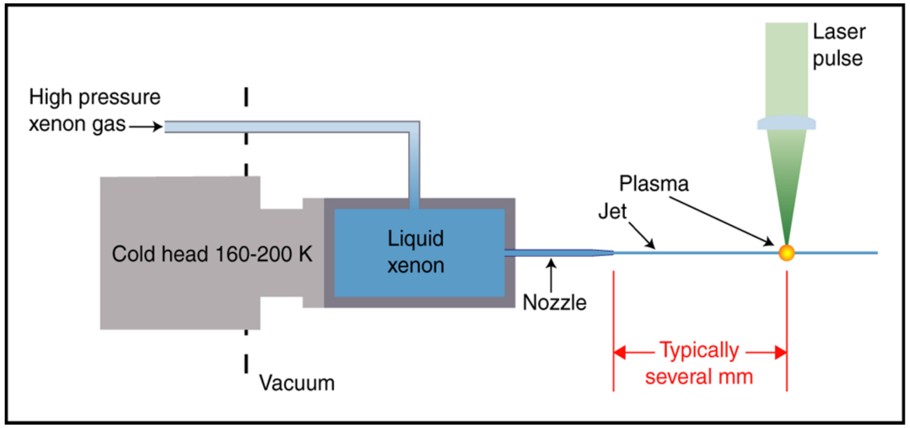
Why use liquid jet/droplet laser-plasmas for HVM EUV litho?

- Thermal
  - Hot plasma at a distance
- High average power via high rep rate
  - Rapid target material production

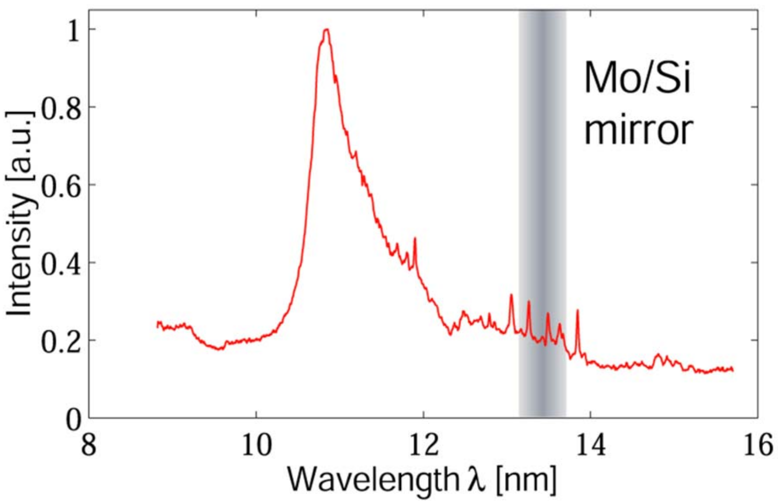
Hertz et al, SPIE (1995)
Malmqvist et al, EUV Litho, OSA (1996)

EUV sources III:
First liquid-xenon-jet source

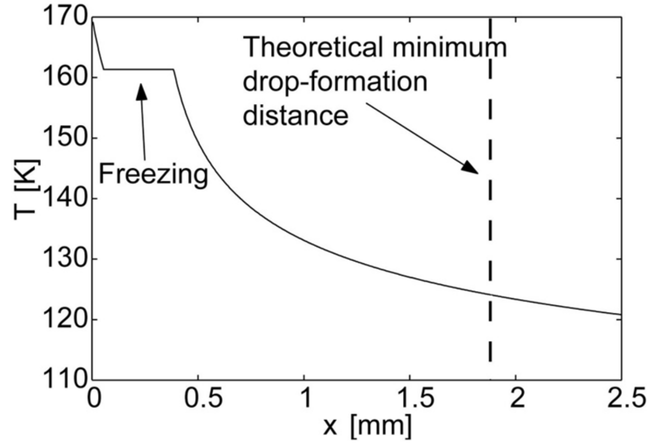
Exp. arrangement



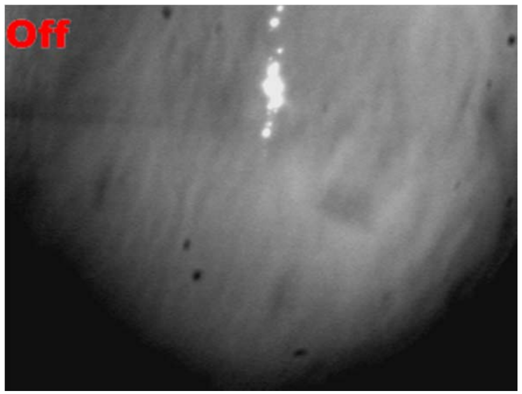
Spectrum

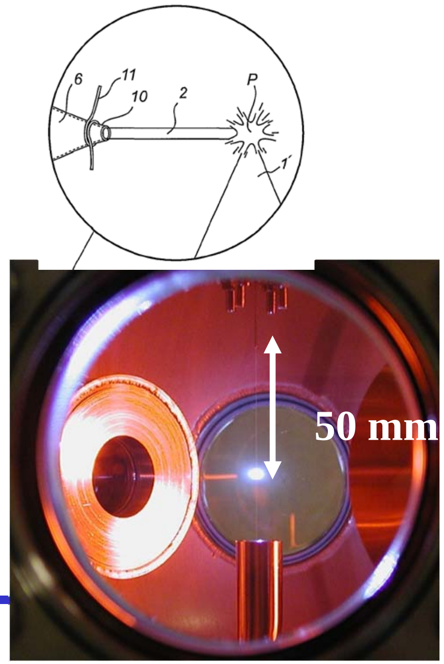


Jet cooling



Stability



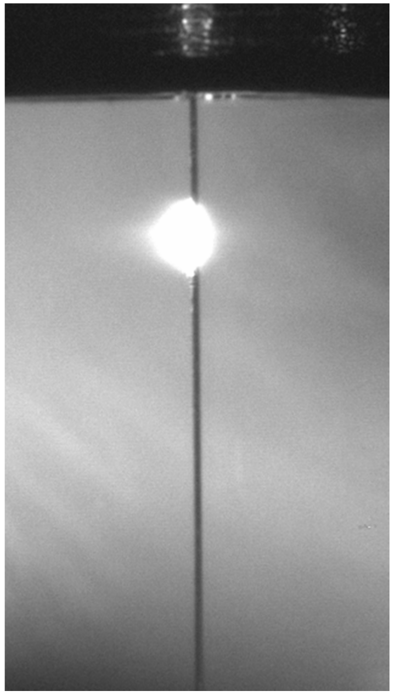


Hansson et al, Microel. Engin. (2000);

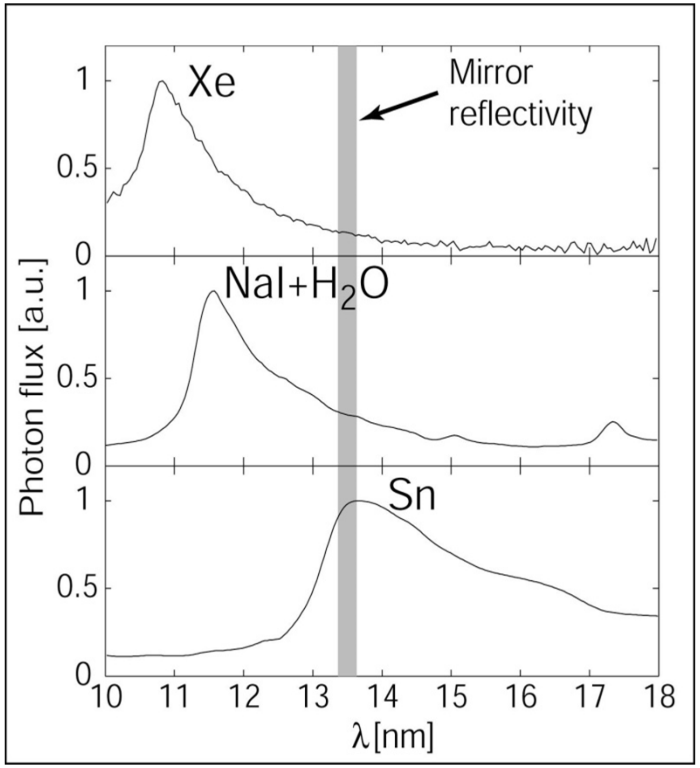
EUV sources IV:

First liquid-tin-jet source

Stable jet @ >250 C



Spectral match



CE: 2.5% into (2%BW×2π×sr)

Debris:

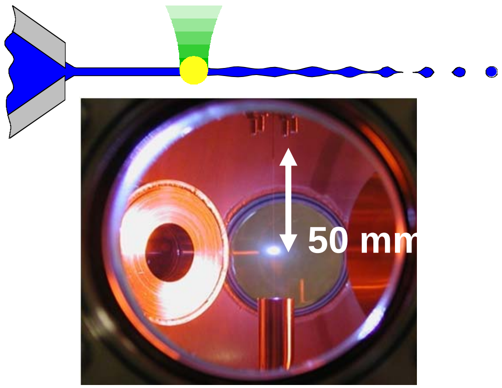
1 h gave coating
Mitigation need: ~10^8

PRESENTLY:
Sn liquid jets prime candidate for HVM EUV litho

Jansson et al .
Appl. Phys. Lett. (2004)

Summary:
Liquid-jet/droplet laser plasmas

Principles



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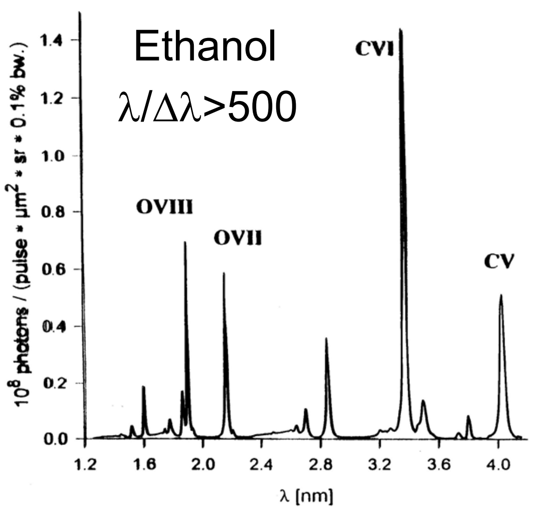
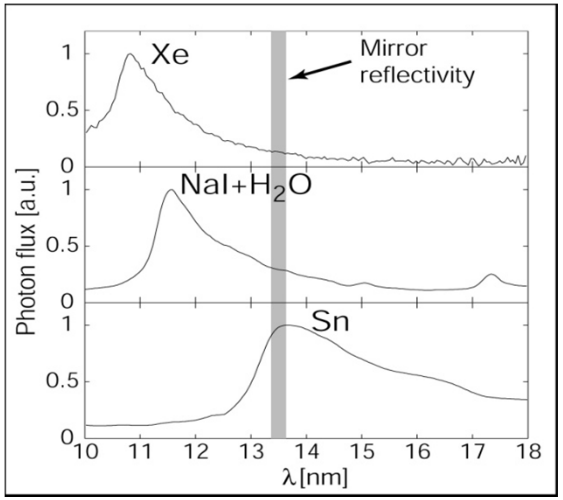
Rymell et al, Opt. Commun. (1993)

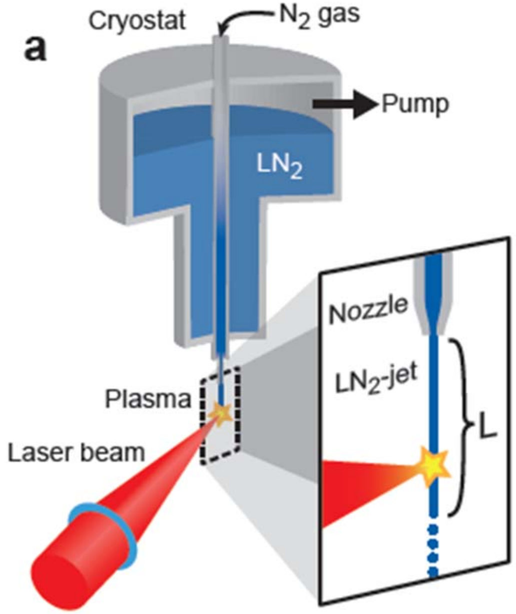
λ≈2-4 nm: Water-window

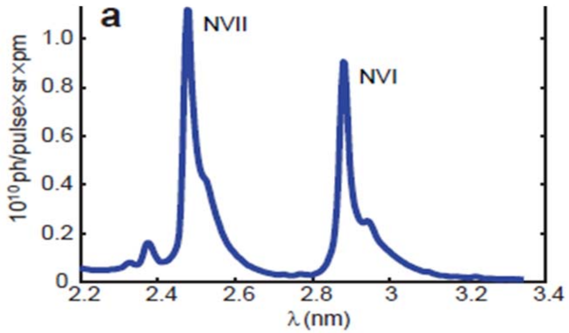


λ=13 nm: EUV Litho



λ≈2.4 nm Liquid nitrogen





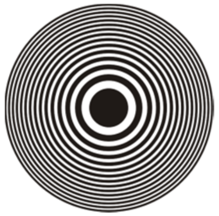
Rymell et al, APL (1995), Berglund et al APL (1997); Jansson et al, RSI (2005) Hansson et al, MNE (2001) ;Takman et al APL (2004) etc

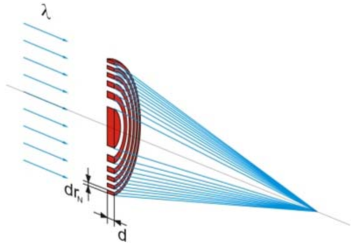
Soft x-ray optics:

Zone plates

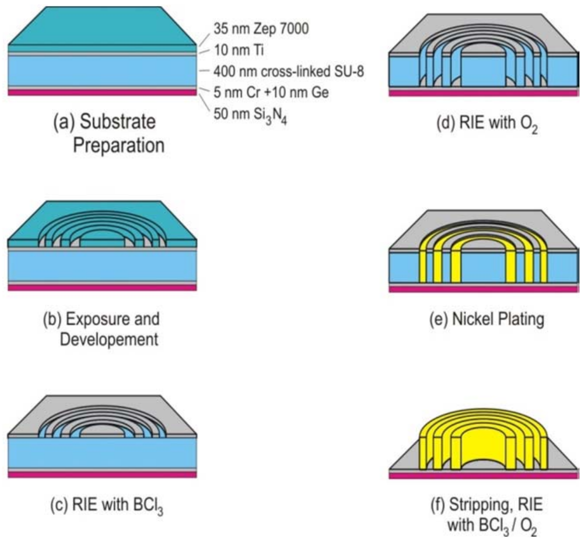
Zone plates:

Circular diffraction gratings

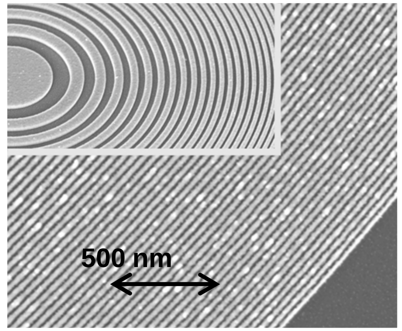




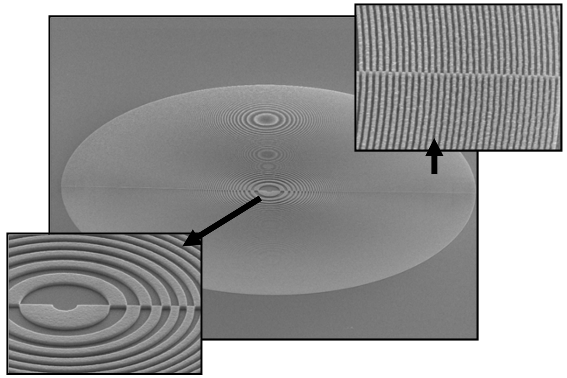
Nano process



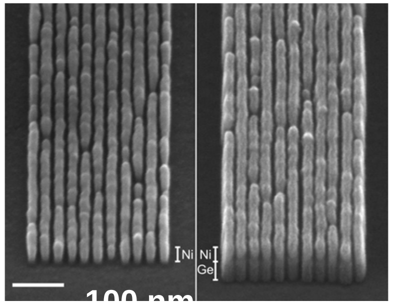
Standard zp:s:



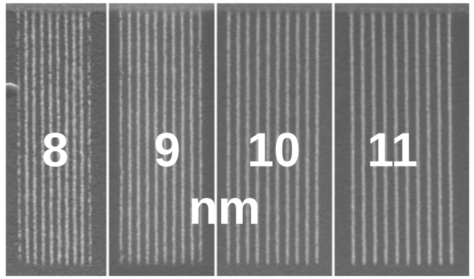
DIC zone plate:

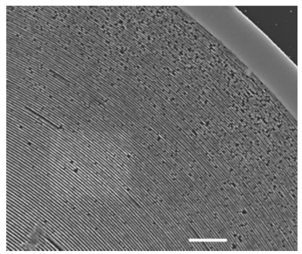


Single-write high resolution:



13 nm Ni-Ge zp





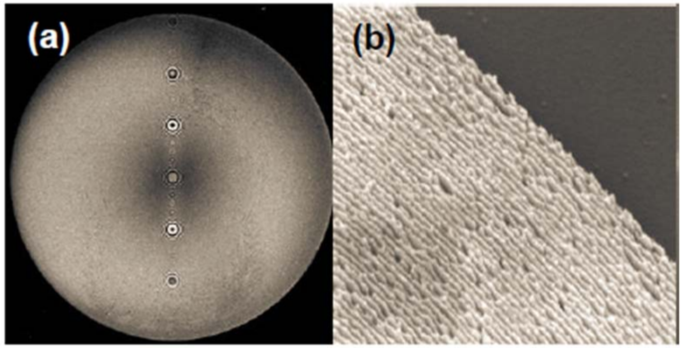
Towards 10 nm Ni and W zp

Reinspach et al, JVST (2009), MNE (2010), JVST (2011) ; in prep (2012); etc

Soft x-ray optics:

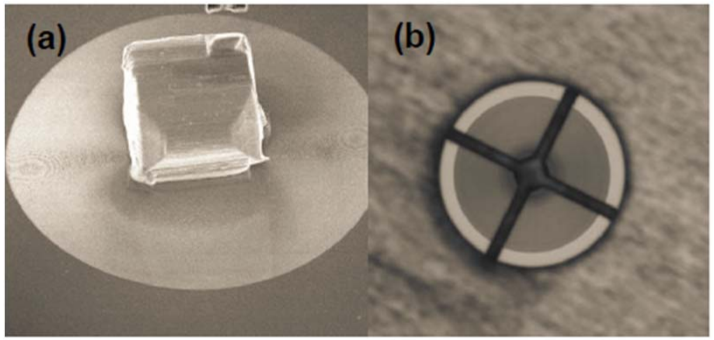
Present x-ray zone plate optics

Ni zp for water-window

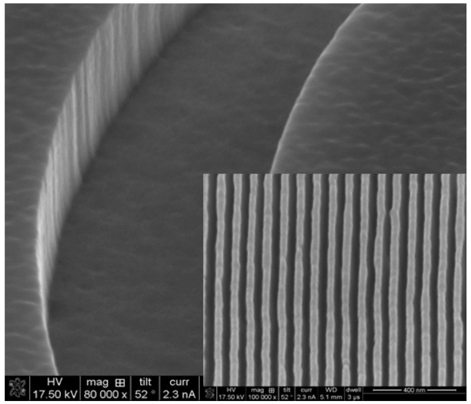


Diam: 200 μm
Outer zone width: 25 nm
Focal length= 2 mm
Circular!

W zp for hard x-rays



W zp with integrated Pt central stop

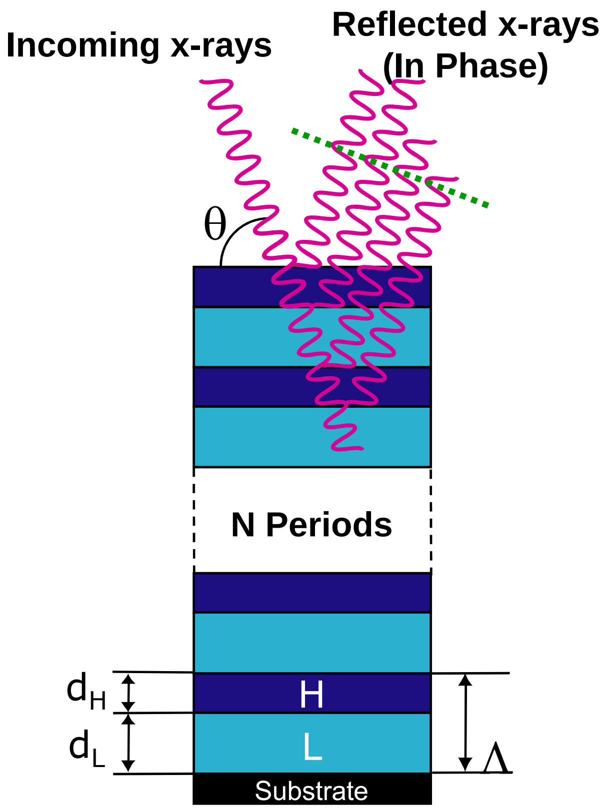


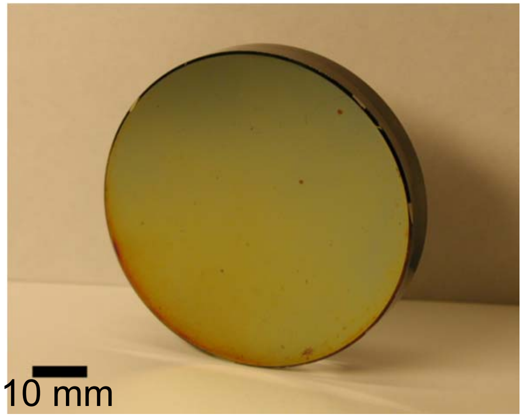
W processing
L/S: 30 nm
Aspect ratio: 20!

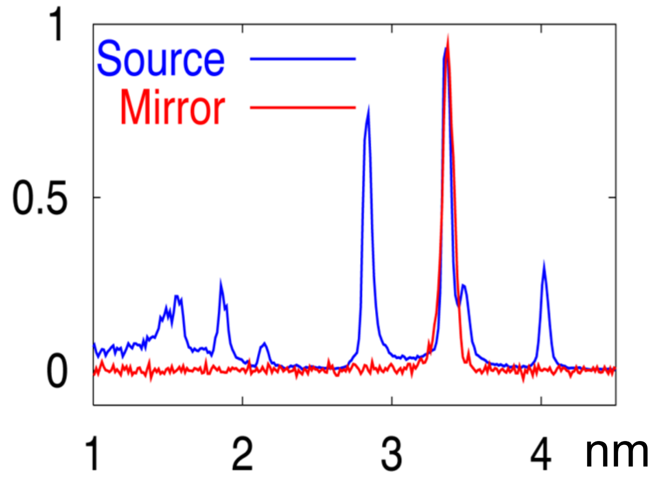
Work of J. Rahomäki and U. Vogt

Soft x-ray optics:

First multilayer condenser optics (1999)







<u>Present mirror:</u>

- Normal incidence
- 200 bi-layers of W/B_4C
- λ = 3.374 nm
- R ~ 0.5 %
- Y. Platonov, Osmic Inc.

<u>Future mirror:</u>

- Normal incidence
- 600 bi-layers of Cr/Sc
- λ = 3.374 nm
- R ~ 5-10 %
- Birch&Eriksson, LiTH

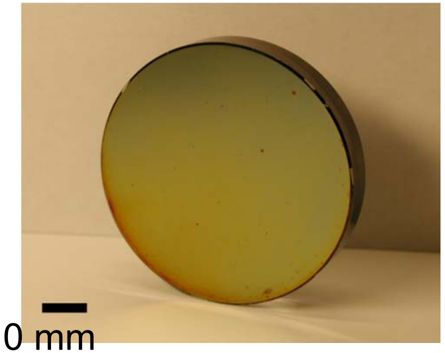
Hertz et al, SPIE (1999).

Soft x-ray optics:

Normal-incidence multilayer condensers (2007)

Present mirrors

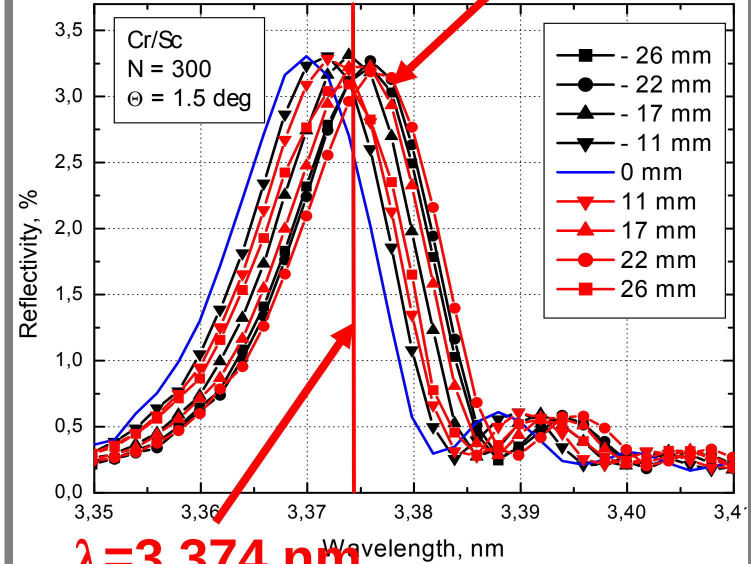
- Cr/Sc
- 58 mm diam
- 350 mm radius
- 300 bilayers
- $\lambda/\Delta\lambda \approx 240$
- Good uniformity
- Good λ match



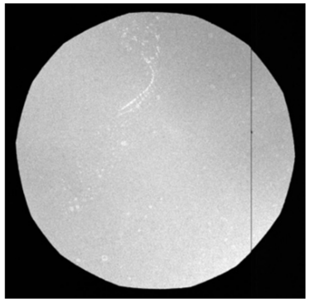
10 mm

Uniformity: Cr/Sc @ 3.374 nm

Refl. >3%!



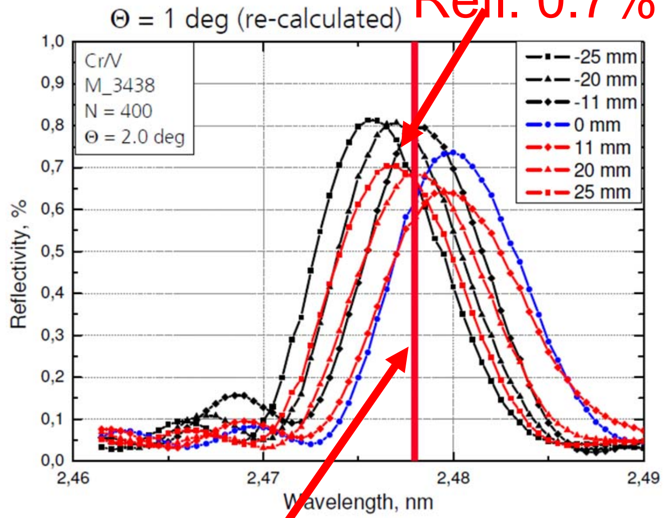
λ=3.374 nm



Stollberg et al, RSI (2007)

Recently: Cr/V@ 2.48 nm

Refl. 0.7%

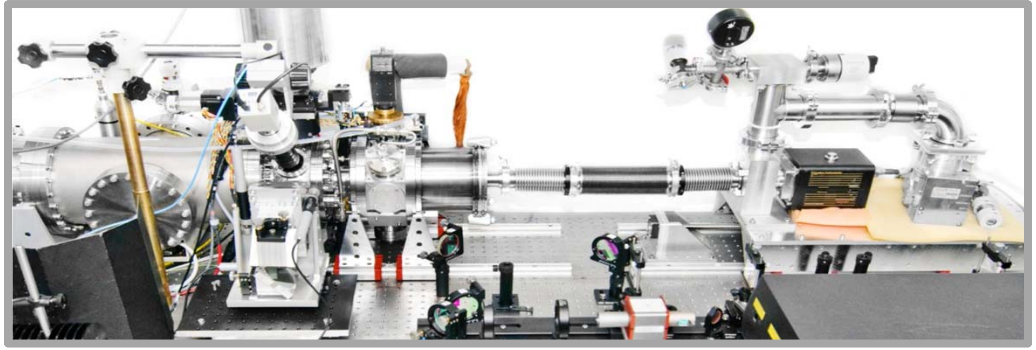


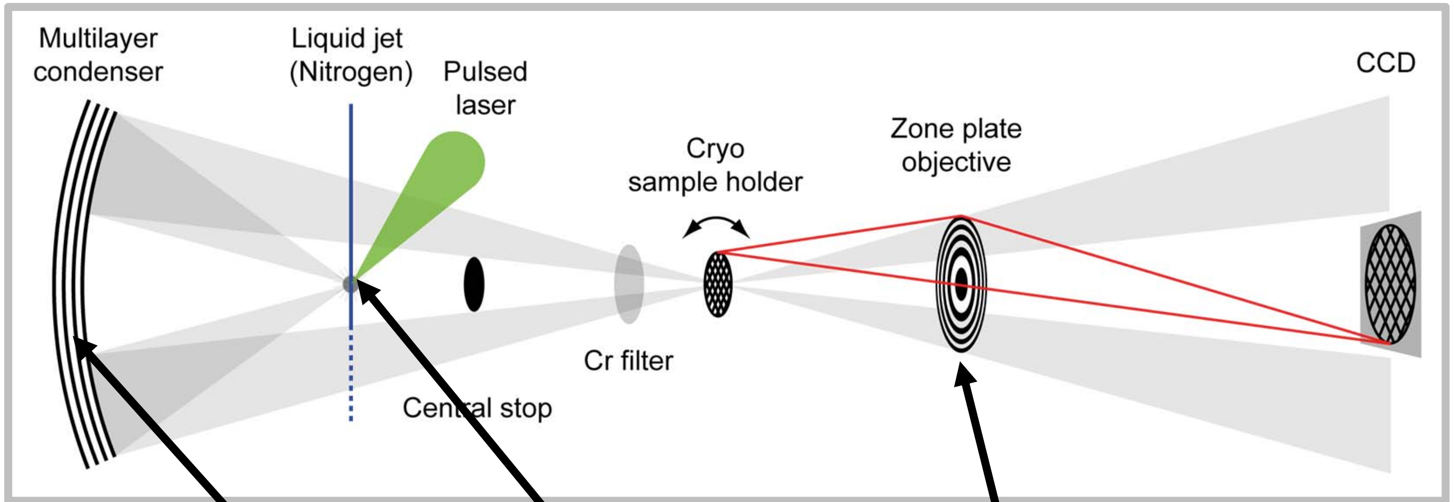
λ=2.478 nm

Takman et al, J. Micro. (2007).

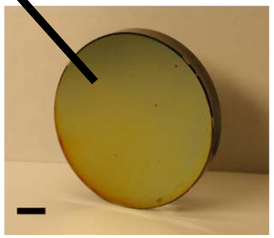
Thanks to S. Yulin, FhG Jena

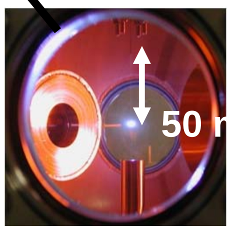
The Stockholm laboratory water-window x-ray microscope

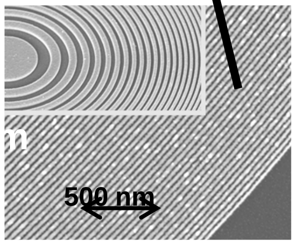




<u>Normal-incidence multilayer mirrors</u> as condensors





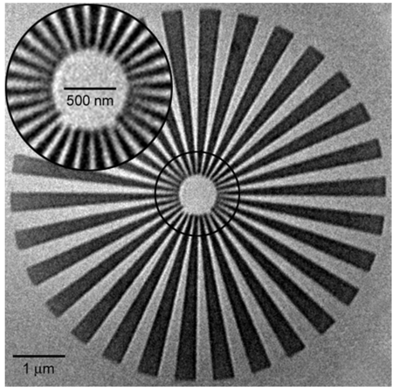


<u>Micro zone plates</u> for high-resolution imaging

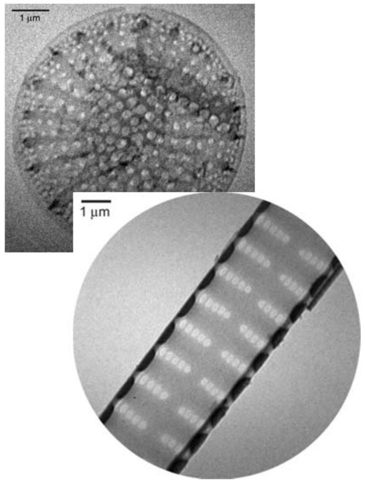
Berglund et al, J. Microsc. (2000), Takman et al, J. Microsc. (2007), Bertilson et al, Opt Expr (2011); Bertilson et al Opt. Lett (2011) etc

Laboratory water-window x-ray microscopy:
2D imaging

Test patterns:

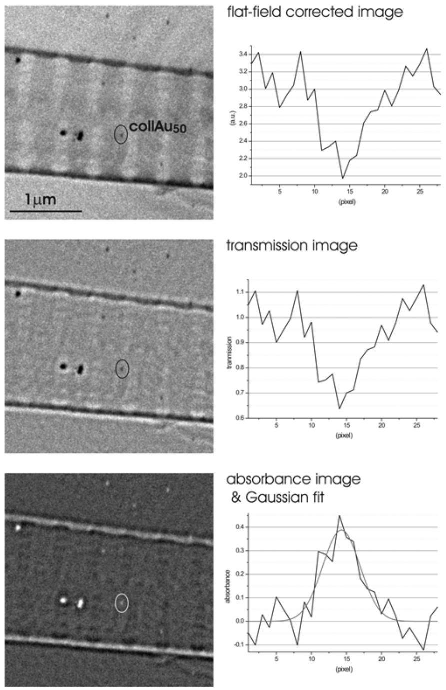


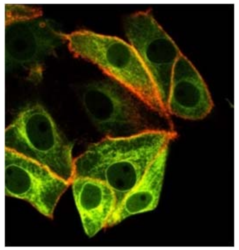
Diatoms:



Function:

Size-selective coll.
Au identification

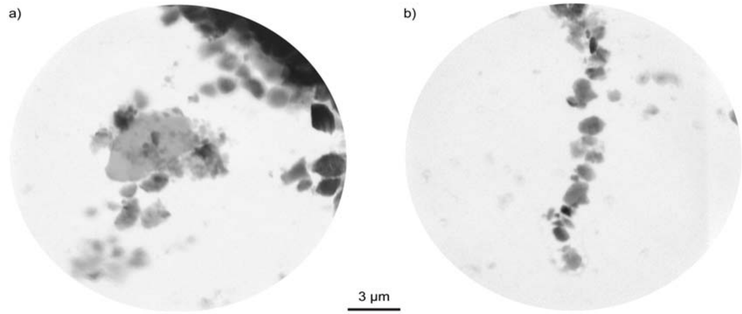




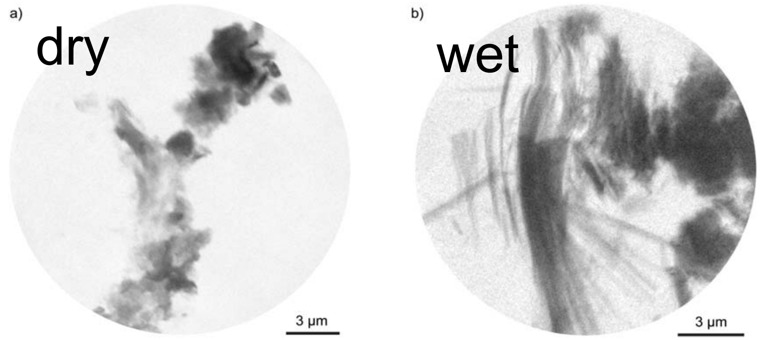
Goal: protein
co-localization

Wet:

Chernozem, wet



Nontronite



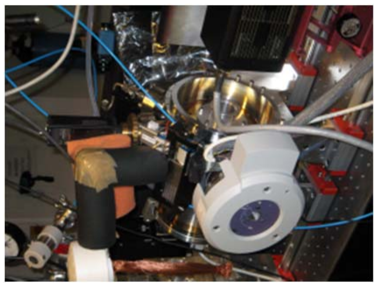
Thanks to J. Thieme, Göttingen/NSLS

Takman et al, J. Microsc. (2007), v. Hofsten et al, Ultramicr (2007) Stollberg et al, J Micr. (2006); Stollberg et al, J Micr. (2007); Hertz et al, Chem Geol (2012)

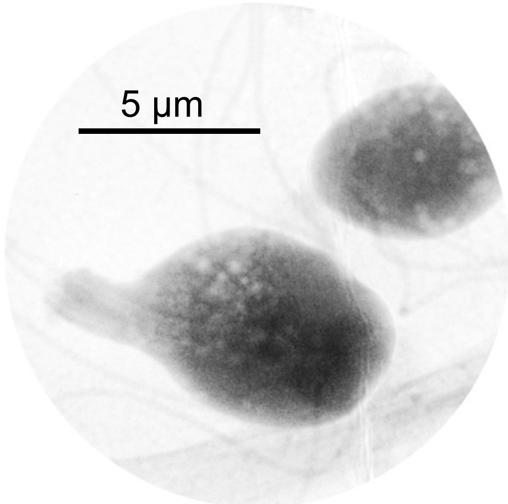
Laboratory water-window x-ray microscopy:

2D cryo imaging

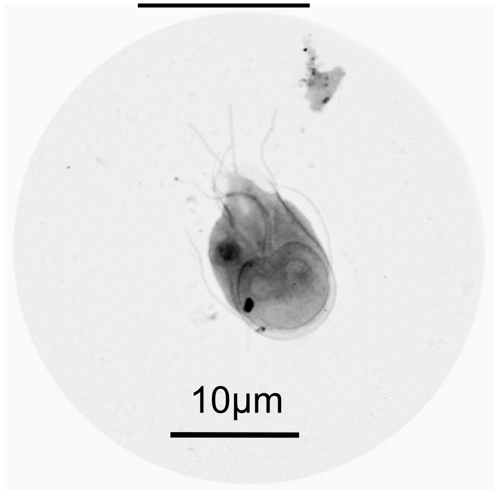
Cryo fixation:



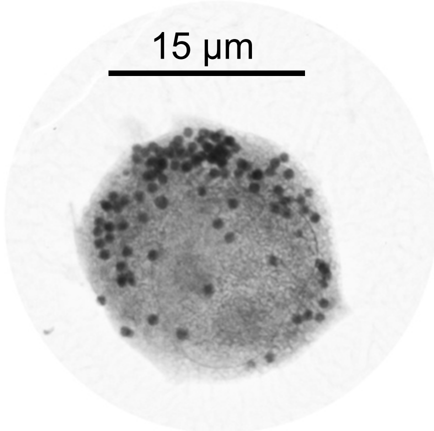
Spironucleus

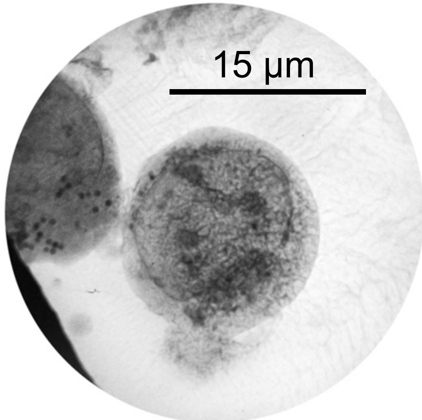


Giardia

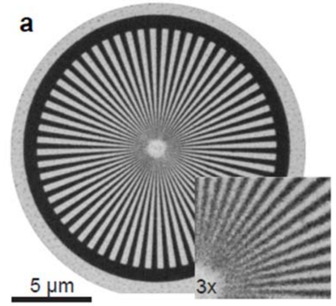


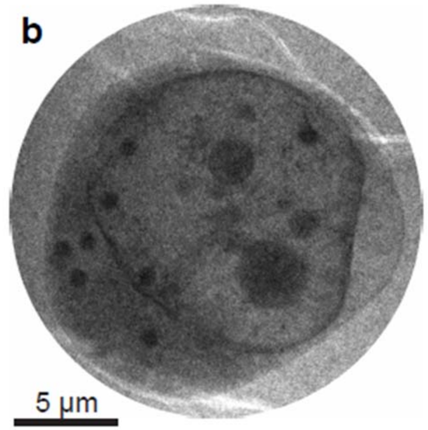
Human immune system B-cells





10 sec exp time!





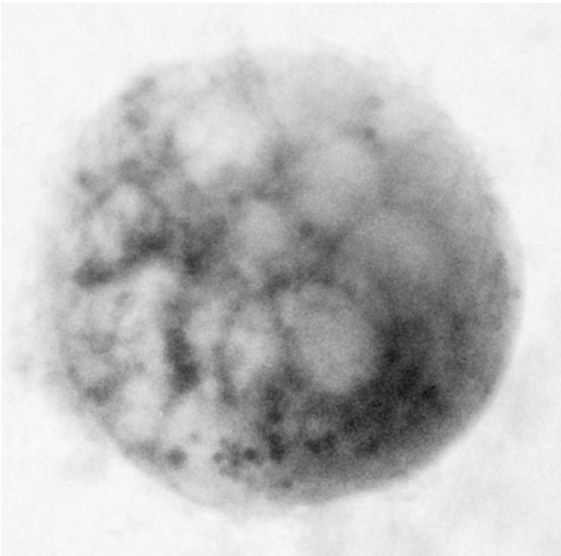
Martz et al, Opt Lett (2012)

Takman et al, J. Microsc. (2007), Bertilson et al, Opt. Lett. (2011); Hertz et al, J. Struct Biol. (2012); Hertz et al, Chem Geol (2012)

Laboratory water-window x-ray microscopy:

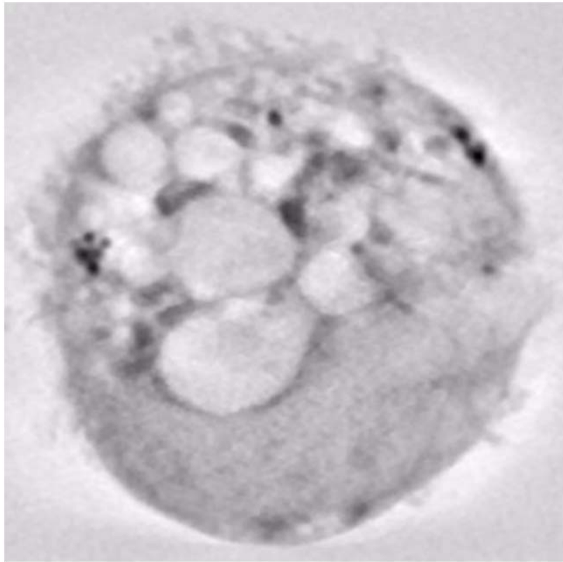
3D: Cryo tomography of human kidney cell

2D image

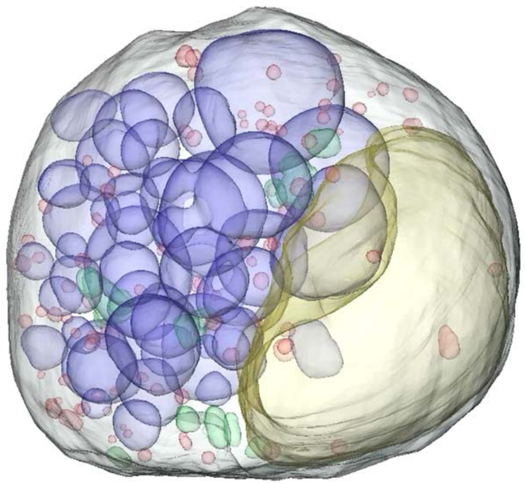


13 μm

Reconstructed volume



Surface rendering



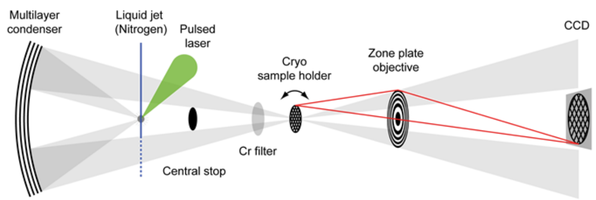
- Tilt series: ±44°, 58 images, 120 s/exp (100 Hz laser)
- Alignment using landmarks
- Reconstruction with SIRT
- Semi-manual segmentation based on local abs coeff.

Bertilsson et al, Opt. Lett (2011); Bertilson et al, Opt. Expr. (2011)

Recent work I:

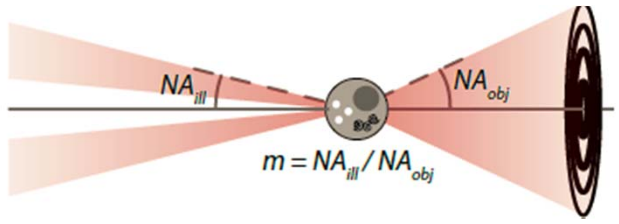
Image formation in soft x-ray microscopes

Experimental arrangement

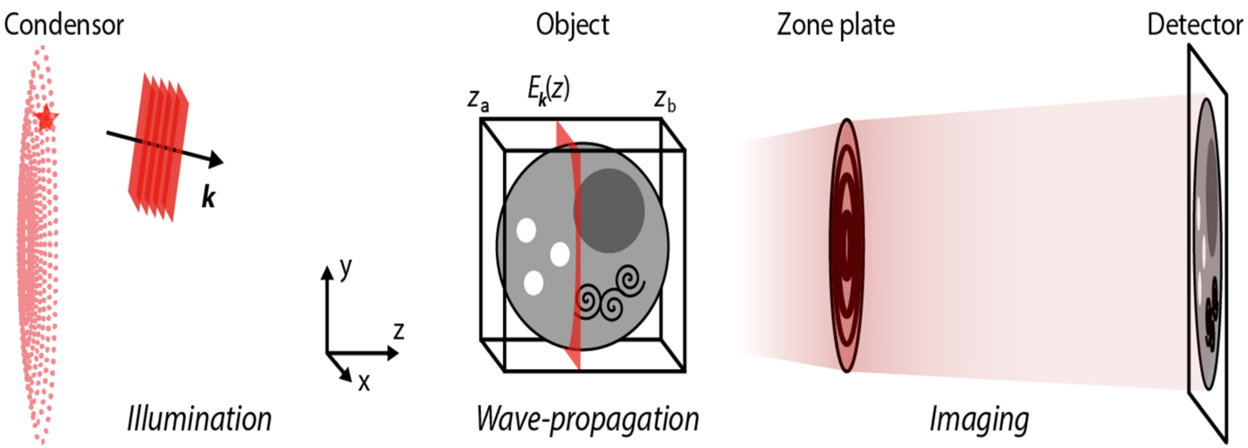


Problem 1: Sample larger than DOF

Problem 2: Partially coherent illumination



3D wave-propagation model



- **3D FDM**
- (10 μm)^3 with 10 nm voxels
- RK4=Runge Kutta
- Parallalizable

- Predicts imaging formation
- Helps interpret images

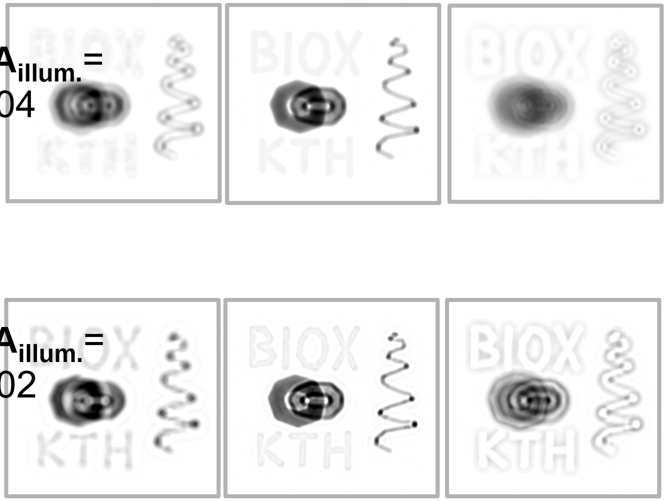
Selin et al, Opt. Expr. (2014)

3D wave-propagation model:

Results

Simulations

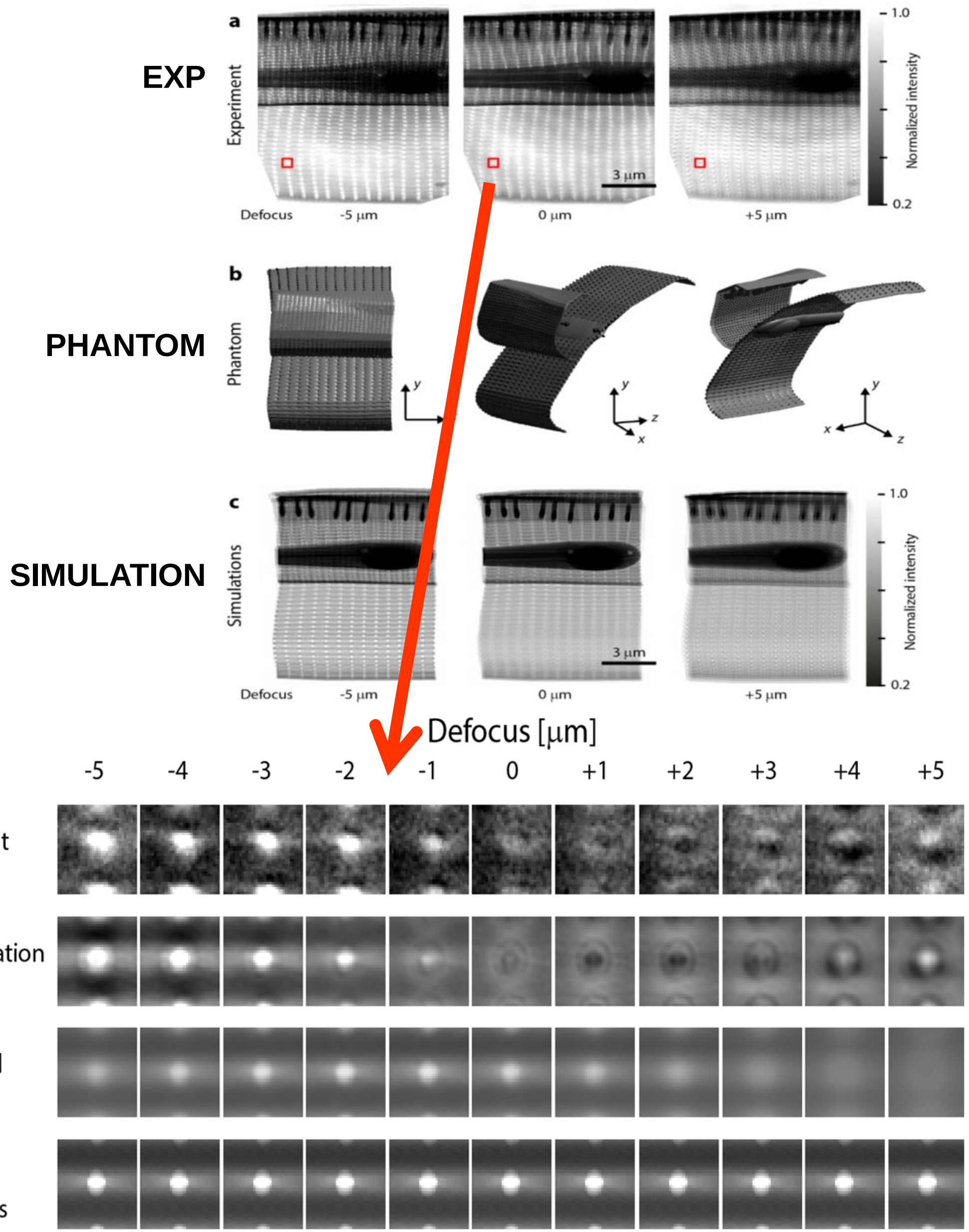
Phantom



-2 µm 0 µm +2 µm

Defocus

Experimental verification @ HZB/Berlin

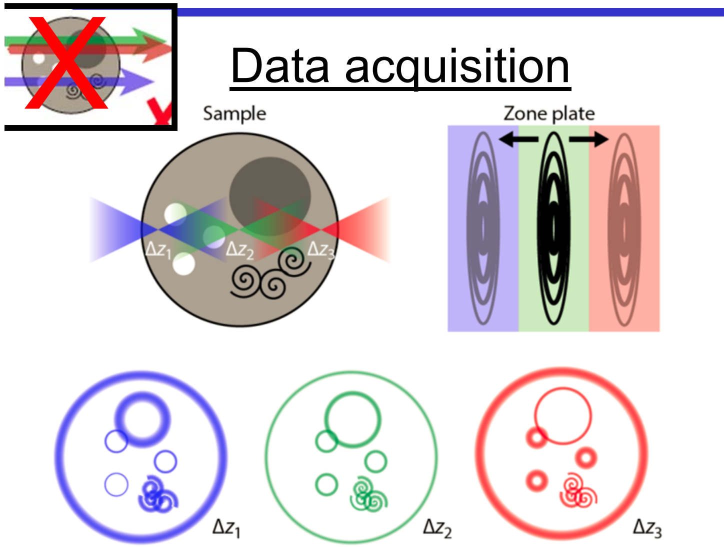


Selin et al, Opt. Expr. (2014)

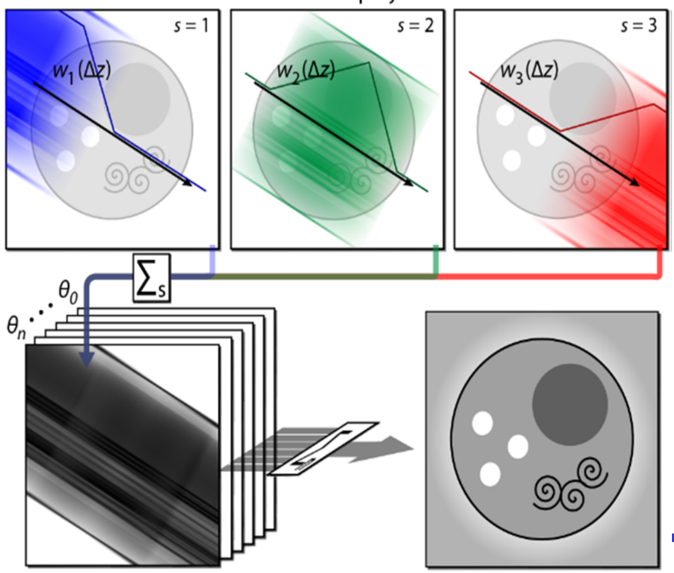
3D with limited DOF:

Tomographic reconstruction with focus-stack back-projection

Data acquisition



Tomographic reconstruction

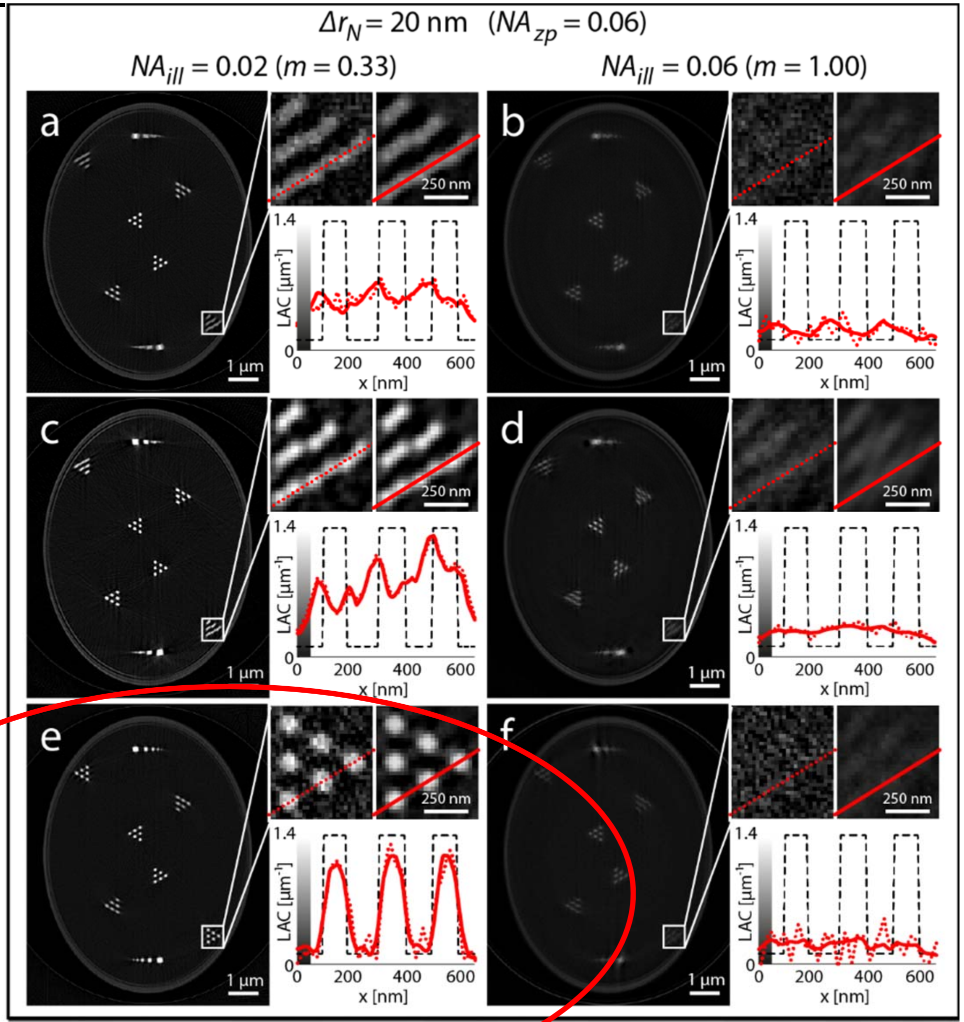


Results

Filtrered BP

Defoc-grad corr BP

Focus-stack BP



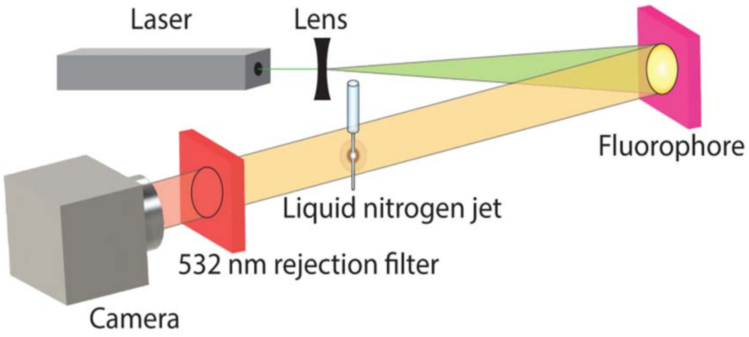
Selin et al, Opt. Lett. (2015)

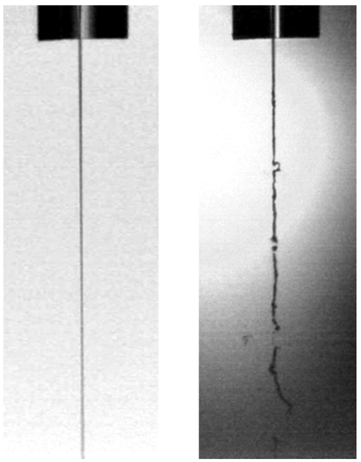
Recent work II:

LN_2 jet laser-plasma stability

High-speed jet imaging

4 ns laser, 20 Hz

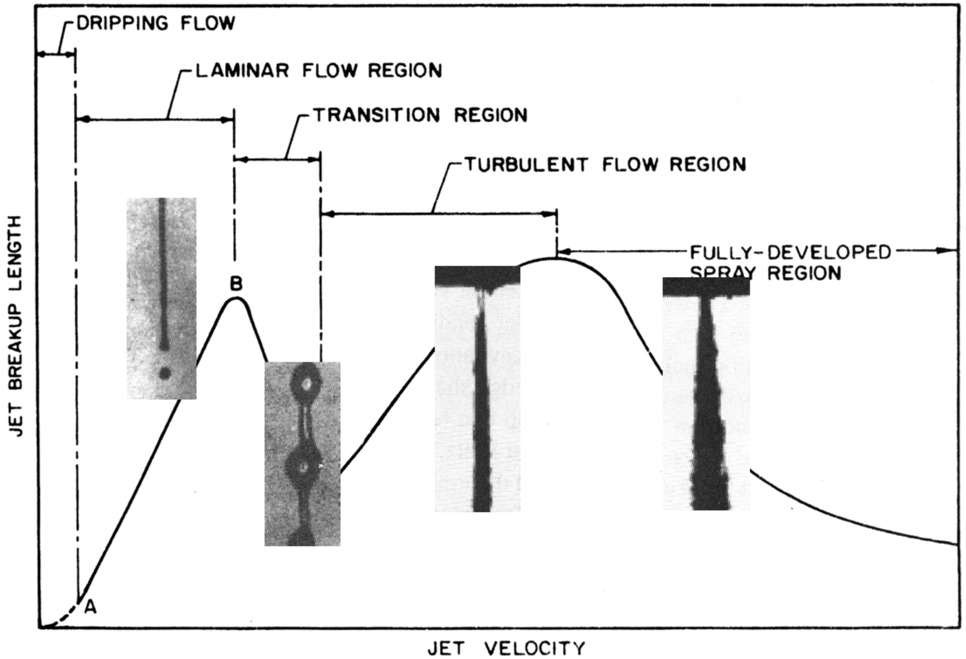




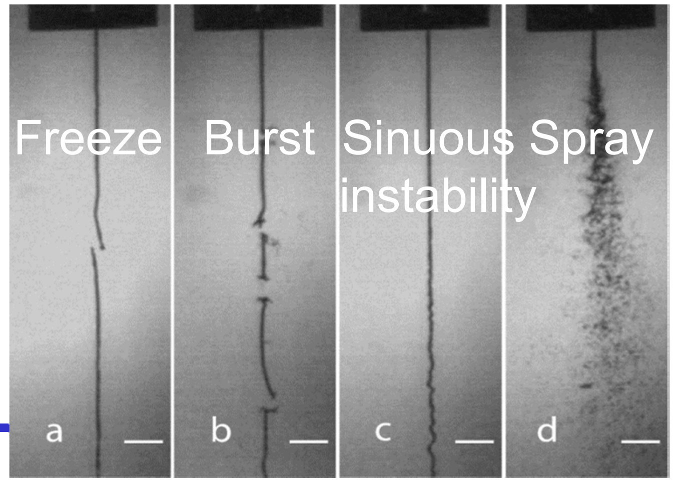
CW Pulsed

Illumination

Classical liquid-jet stability



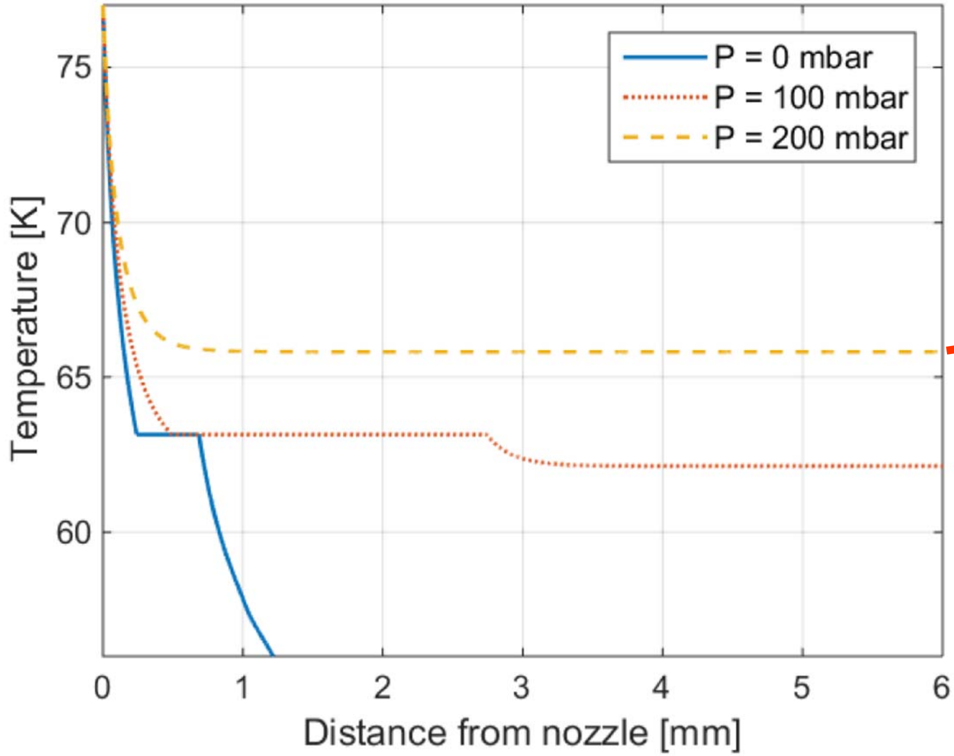
LN2 liquid-jet stability



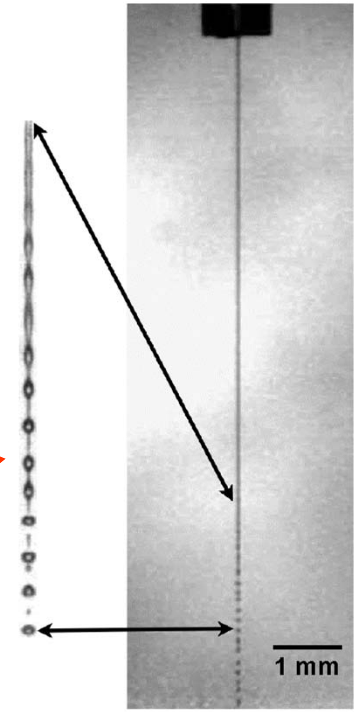
Fogelqvist et al, J. Appl. Phys. (2015)

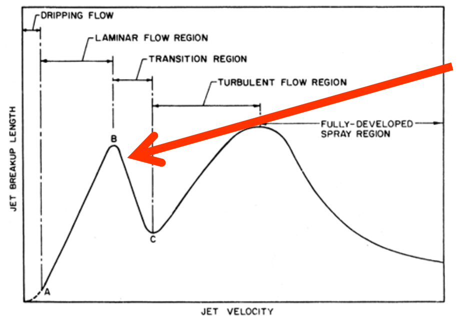
Why?
Evaporation effects

Jet temperature vs surrounding pressure



STABLE!





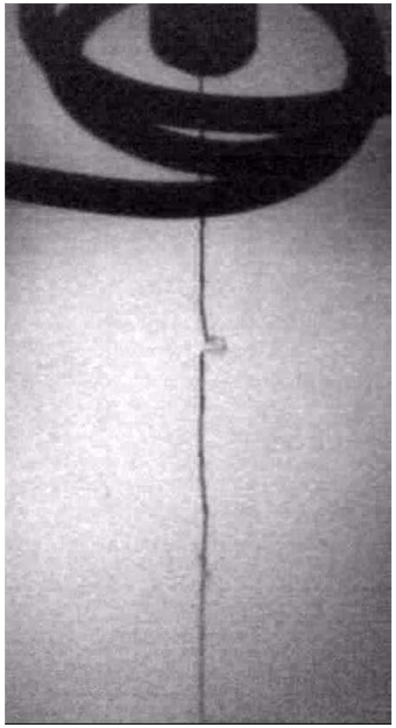
Counter-intuitive!

We cannot use Pressure!

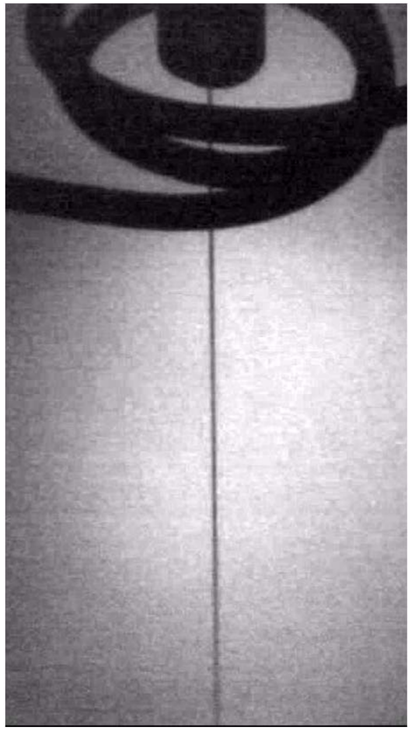
Fogelqvist et al, J. Appl. Phys. (2015)

Avoiding evaporative effects while still keeping pressure low

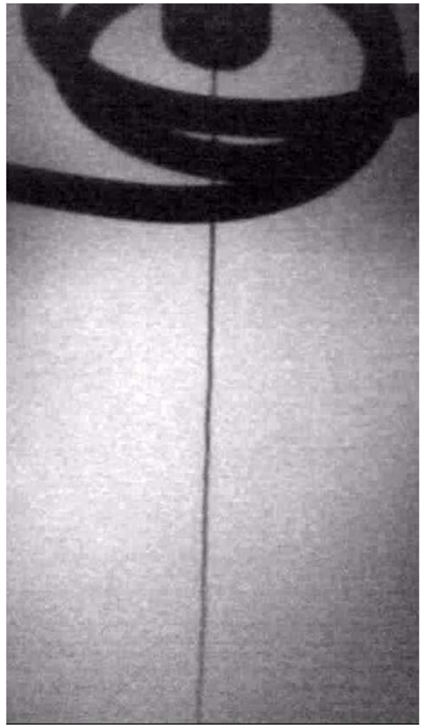
<u>Local radiative heating</u>



No stabilization



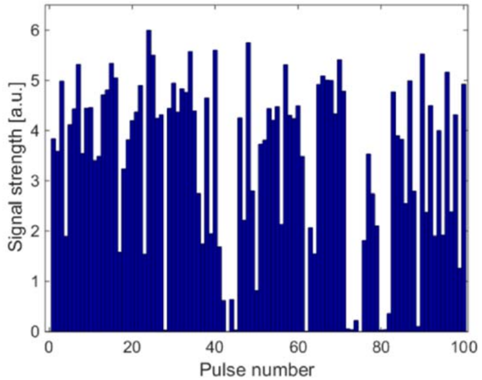
Pressure stabilization



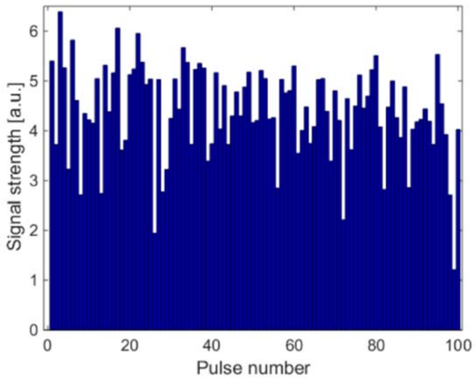
Radiative heat stabilization

Results:
Improved laser-plasma stability

No heating

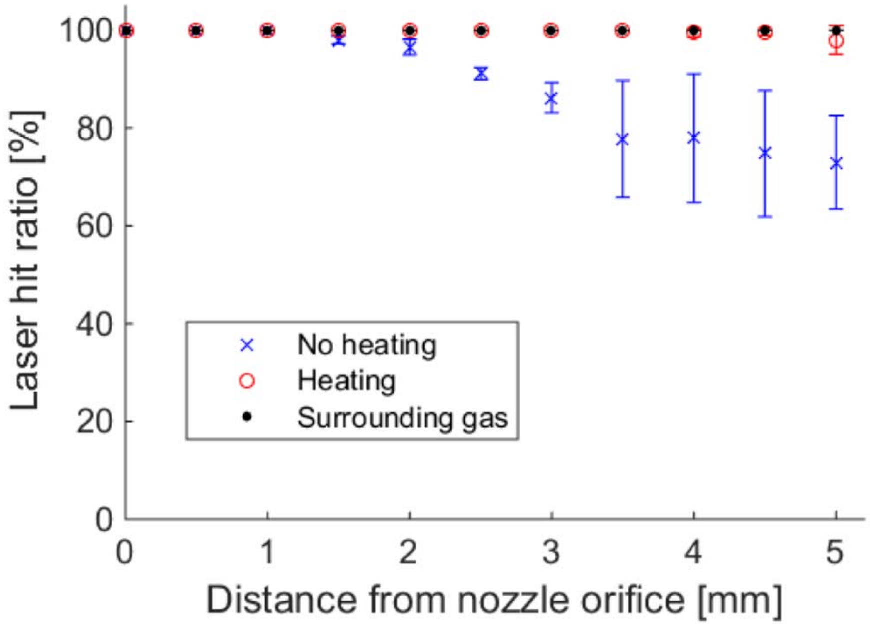


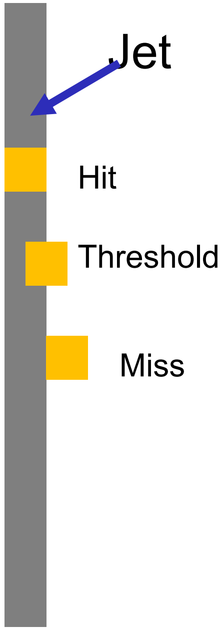
With heating



Quantitative improvment:

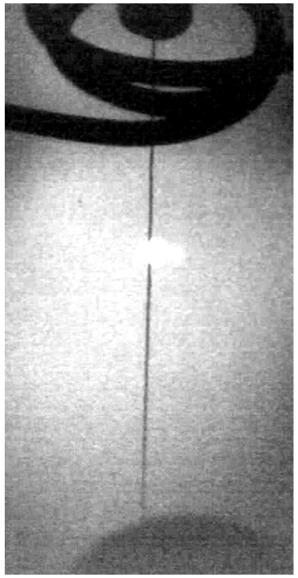
Hit ratio from movies

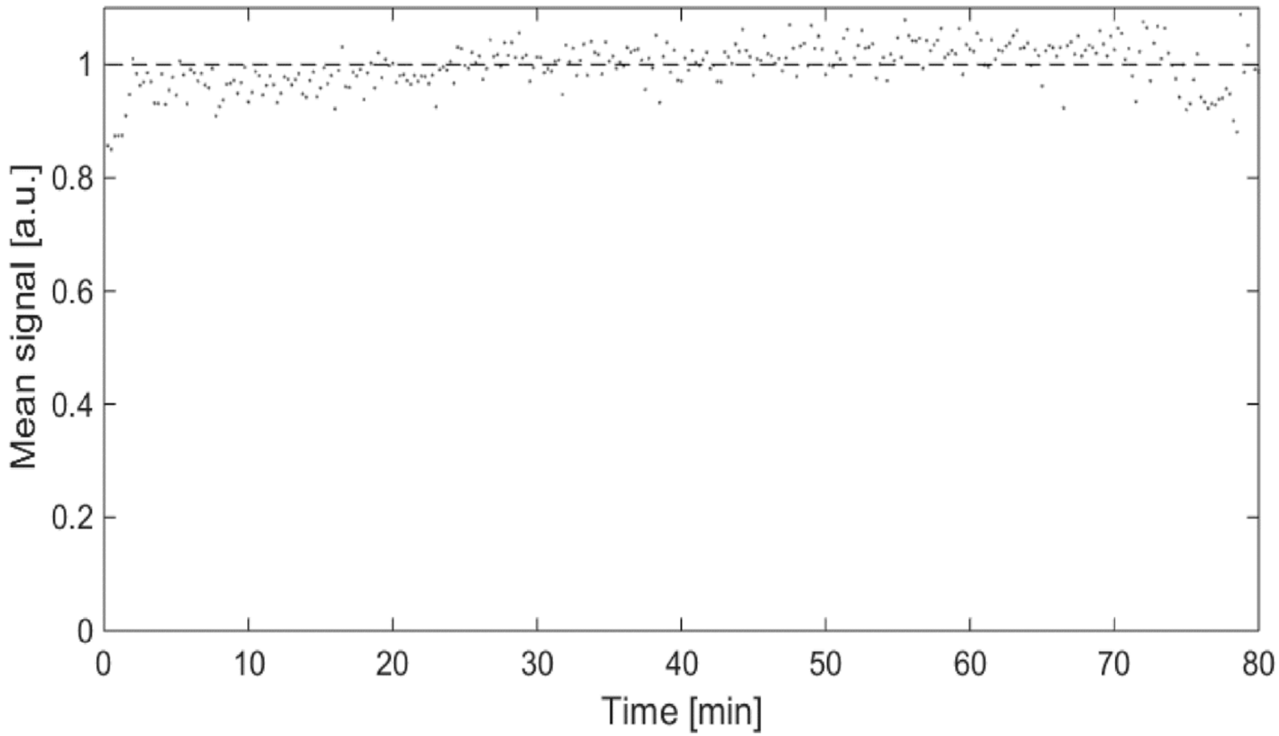




Fogelqvist et al, J. Appl. Phys. (2015)

Long-term stability: 1h



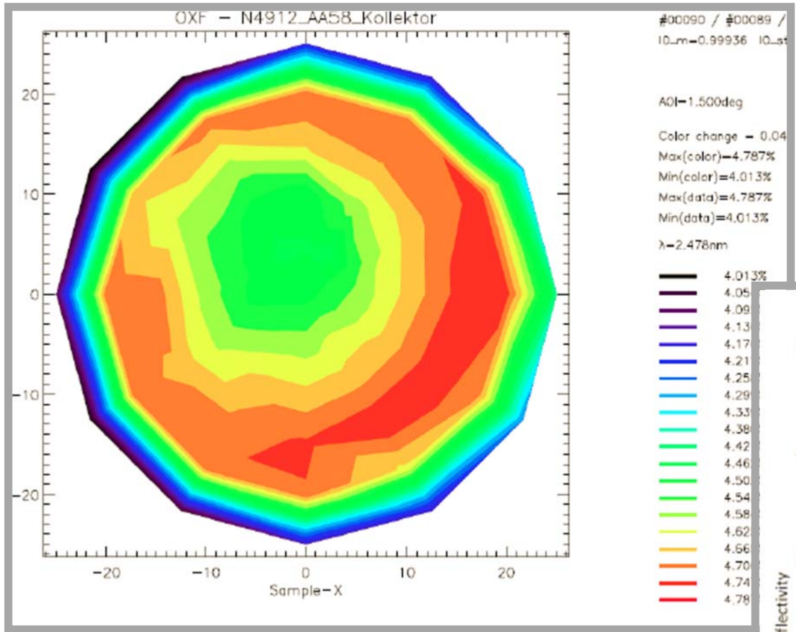


OK for tomography

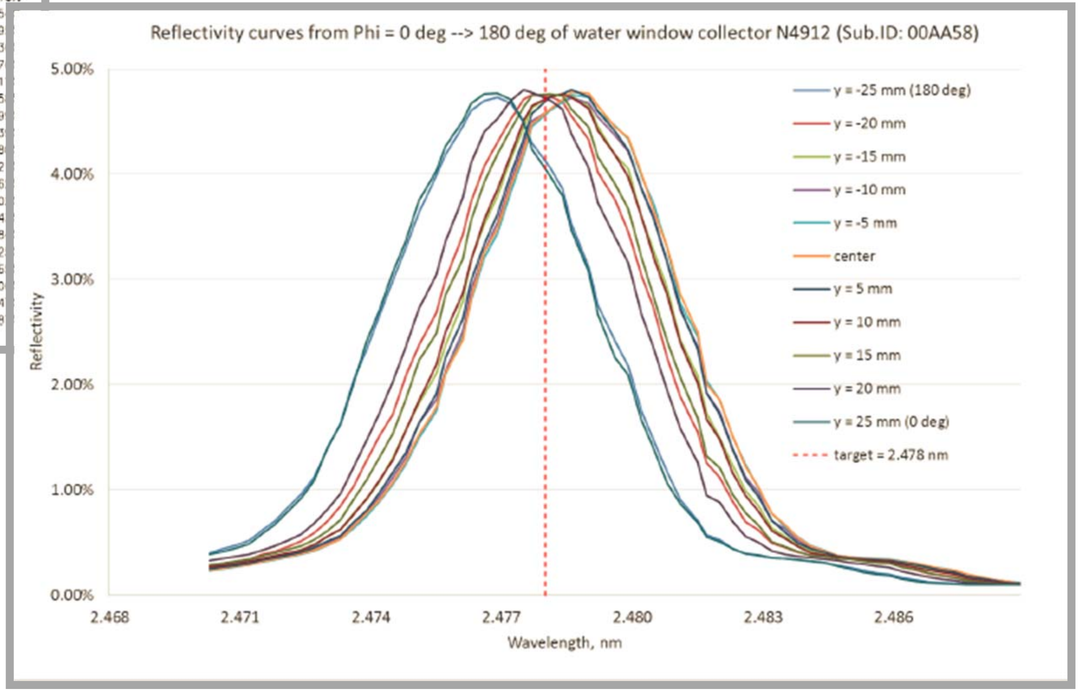
Fogelqvist et al, J. Appl. Phys. (2015)

Recent work III:

New ML mirrors



R>4% @ λ=2.48 nm



Thanks: Torsten Feigl et al, Jena

Summary

- Laboratory soft x-ray 3D microscopy
  - Liquid-nitrogen-jet laser-plasma sources
    - Early bending-magnet brightness
    - Improved long-term stability
  - 3D imaging
    - High-resolution 3D microscopy of intact cells
    - New model for taking DOF and partial coherence into account
    - New tomographic routine: focus-stacked backprojection
    - 10 s per projection exposure
  - Next
    - Stable long-term data acquisition
    - Implement new tomo algorithm on bio cells
    - Optimize illumination for contrast
    - Cell-cell interactions
    - Virus

Biomedical & X-Ray Physics

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